

10/785, 229

EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L9	1964	502/209 OR 502/305 OR 502/306 OR 502/321 OR 560/231 OR 560/241	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	OFF	2006/03/22 06:22
L10	1071	L9 AND (CARBOXYLIC OR CARBOXYLIC OR CARBOXYLATE OR ACETIC OR PROPIONIC)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L11	974	L10 AND (CATALYST OR HETEROPOLYACID OR TUNGSTATE OR MOLYBDATE OR VANADATE OR SILICOTUNGSTATE OR PHOSPHOVANADATE OR PHOSPHOMOLYBDATE OR PHOSPHOTUNGSTATE OR SILICOPHOSPHOTUNGSTATE OR SILICOPHOSPHOVANADATE OR SILICOPHOSPHOMOLYBDATE OR SILICOPHOSPHOVANDIC OR SILICOPHOSPHOMOLYBDIC OR SILICOPHOSPHOTUNGSTIC OR TUNGSTIC OR MOLYBDIC OR VANDADIC OR SILICOTUNGSTIC OR PHOSPHOVANDADIC OR PHOSPHOMOLYBDIC)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L12	330	L11 AND (OLEFIN OR OLEFINE)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L13	298	L12 AND (GAS OR VAPOR OR VAPOUR)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L14	226	L13 AND (SUPPORT OR SUPPORTED OR SUPPORTING)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L15	221	L14 AND (REACTOR OR COLUMN OR COLUMNAR OR VESSEL OR APPARATUS OR APPARATTUS OR APARATTUS)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22

EAST Search History

10/785, 229

L16	143	L15 AND CONTACTING	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:22
L17	6	I16 and (lower adj aliphatic adj carboxylic)	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:23
L18	54	I16 and ester	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 08:51
L19	0	I18 not I18	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:23
L20	48	I18 not I17	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 06:23
L21	10	I9 and (lower adj (olefin or olefine)) and ((carboxylic adj acid adj ester) or (carboxylic adj ester)) and ((gas adj phase) or ((vapor or vapour) adj phase)) <i>CLASSE + SVB CLASSED (ON PREV. PAGE) & VERY</i>	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2006/03/22 08:53

22 ACETICS

221358 ACETIC
(ACETIC OR ACETICS)

54670 PROPONICS

8 PROPONICS
54674 PROPONICS
258085 PROPONICS
(PROFONIC OR PROPONICS)

258085 ACRYLICS

1322 ACRYLICS

258457 ACRYLIC
(ACRYLIC OR ACRYLICS)

73789 METHACRYLIC
6 METHACRYLICS

73793 METHACRYLIC
(METHACRYLIC OR METHACRYLICS)

L1 ANSWER 1 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 20041036764 CAPLUS
DOCUMENT NUMBER: 142 40422
TITLE: Support and catalyst for use in producing lower
aliphatic carboxylic acid ester,
catalyst manufacture process and method of use
Kadowaki, Etsuko; Higashi, Tomoyoshi; Oguchi, Wataru;
Uchida, Hiroshi; Naumi, Kousuke
Showa Denko K.K., Japan
U.S. Pat. Appl. Publ., 27 PP., Cont.-in-part of U.S.
Ser. No. 70-259, abandoned.
Coden: USXKC0

=> D 1-5 IBIB ABS

L1 ANSWER 1 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 20041036764 CAPLUS

DOCUMENT NUMBER: 142 40422

TITLE: Support and catalyst for use in producing lower
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catalyst manufacture process and method of use

Kadowaki, Etsuko; Higashi, Tomoyoshi; Oguchi, Wataru;

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Showa Denko K.K., Japan

U.S. Pat. Appl. Publ., 27 PP., Cont.-in-part of U.S.

Coden: USXKC0

Patent

English

3 FAMILY PCC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.: -----

KIND: -----

DATE: -----

APPLICATION NO.: -----

DATE: -----

US 2003118497 A1 20030626 US 2002-70259
ZA 2002009804 A 20030819 ZA 2002-9804
ZA 2003005143 A 20040312 A 2003-5143
PRIORITY APPLN. INFO.:

US 2002009804 P 20020304
ZA 20021203 P 20021203
JP 2000-192964 A 20000627
US 2000-218803P P 20000718
JP 2001-35038 A 20010213
US 2001-273343P P 20010306
WO 2001-35532 W 20010627
US 2001-389354 B2 20010717
JP 2001-313675 A 20011207
WO 2002-30156 W 20020212
US 2002-70259 B2 20020304

OTHER SOURCE(S):
AB: The catalyst is produced by a process comprising a step of contacting the catalyst (hetropolycarboxylic salts or their salts) with a gas containing at least one member selected from water, lower aliphatic carboxylic acids and lower aliphatic alics. The catalyst can exhibit high initial activity and high space time yield, ensure sufficiently long catalyst life in practice and can prevent the production of byproduct materials. A siliceous support is provided for a catalyst, which has a Si content of 39.7-46.3% or a Si content of 85.99% in terms of silicon dioxide or a crush strength of 30 N or more. By the use of a catalyst comprising the support, a lower aliphatic carboxylic acid ester is produced from lower olefin and a lower aliphatic carboxylic acid in a gas phase without causing great reduction of catalytic activity or cracking or abrasion of the catalyst.

CASRACT 142:40422
L1 ANSWER 2 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 20031006934 CAPLUS
DOCUMENT NUMBER: 140:43772
TITLE: Process and catalysts for the production of aliphatic carboxylic acid esters from lower aliphatic carboxylic acids and lower olefins
Watanabe, Kyoichi; Uchida, Hiroshi
Showa Denko K. K., Japan
PCT Int. Appl., 30 pp.

L1 ANSWER 2 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 20031006934 CAPLUS
DOCUMENT NUMBER: 140:43772
TITLE: Process and catalysts for the production of aliphatic carboxylic acid esters from lower aliphatic carboxylic acids and lower olefins
Watanabe, Kyoichi; Uchida, Hiroshi
Showa Denko K. K., Japan
PCT Int. Appl., 30 pp.

INVENTOR(S):
PATENT ASSIGNEE(S):
SOURCE:
DOCUMENT TYPE:
LANGUAGE:
FAMILY ACC. NUM. COUNT:
PATENT INFORMATION:

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PATENT INFORMATION:

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PATENT ASSIGNEE(S):
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DOCUMENT TYPE:
LANGUAGE:
FAMILY ACC. NUM. COUNT:
PATENT INFORMATION:

INVENTOR(S):
PATENT ASSIGNEE(S):
SOURCE:
DOCUMENT TYPE:
LANGUAGE:
FAMILY ACC. NUM. COUNT:
PATENT INFORMATION:

22 ACETICS

221358 ACETIC
(ACETIC OR ACETICS)

54670 PROPONICS

8 PROPONICS
54674 PROPONICS
258085 PROPONICS
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258085 ACRYLICS

1322 ACRYLICS

258457 ACRYLIC
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73789 METHACRYLIC
6 METHACRYLICS

73793 METHACRYLIC
(METHACRYLIC OR METHACRYLICS)

L1 ANSWER 1 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 20041036764 CAPLUS
DOCUMENT NUMBER: 142 40422
TITLE: Support and catalyst for use in producing lower
aliphatic carboxylic acid ester,
catalyst manufacture process and method of use

Kadowaki, Etsuko; Higashi, Tomoyoshi; Oguchi, Wataru;

Uchida, Hiroshi; Naumi, Kousuke

Showa Denko K.K., Japan

U.S. Pat. Appl. Publ., 27 PP., Cont.-in-part of U.S.

Coden: USXKC0

Patent

English

3 FAMILY PCC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.: -----

KIND: -----

DATE: -----

APPLICATION NO.: -----

DATE: -----

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WO 2002-JP11683 W 20021108
 WO 2002-JP11686 W 20021108

OTHER SOURCE(S): CASREACT 140:43772
 AB Lower aliphatic carboxylic acid esters (e.g., Et acetate) are prepared by esterifying a lower aliphatic carboxylic acid and a lower olefin (e.g., ethylene) into a lower aliphatic carboxylic acid ester using an acid catalyst in a vapor phase, when the system is controlled to contain substantially no acetylene compds., the deterioration of the catalyst can be remarkably prevented from proceeding and in turn a stable operation can be continuously performed for long time. Examples of the acid catalyst which can be used in the present invention include compds. widely known in general as an acid catalyst, such as a heteropolyacid and a salt; process flow diagrams are presented.

REFERENCE COUNT: 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L1 ANSWER 3 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 2000:88415 CAPLUS
 DOCUMENT NUMBER: 132:94968
 TITLE: Process and catalysts for the manufacture of esters by the addition reaction of lower alkenes with aliphatic monocarboxylic acids with removal of nitrogenous bases from the reactants

INVENTOR(S): Coker, Eric Nicholas; Froom, Simon Frederick Thomas; Smith, Warren John
 BP Chemicals Limited, UK
 PCT Int. Appl., 16 pp.
 CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 1999-0127	WO	1999-0701	19990701	
WO 1999-02099	WO	1999-0701	19990701	
WO 1999-03966	AI	2000-0127	19990701	
CA 2336946	AI	2000-0127	19990701	
AU 9946323	AA	2000-0127	19990701	
BR 9912038	A1	20010403	1999-12038	19990701
EP 1097120	A1	20010509	EP 1999-92533	19990701
R: AV, BE, CH, DE, ES, FR, GR, IT, LI, LU, NL, SE, BF, BJ, CF, CG, CI, CM, GA, SN, TD, TS				
CA 1999-2336946				
BR 19990207	AU 1999-46323			
EP 20000207	BR 1999-12038			
JP 2002250380	T2	20020709	JP 2000-560076	19990701
TW 502016	B	20020911	TW 1999-88111867	19990713
US 2001047107	A1	20011129	US 2001-752835	20010103
ZA 2001000331	A	20020211	ZA 2001-331	20010111
PRIORITY APPLN. INFO.:				

AB Lower aliphatic esters are prepared in high yield and selectivity by the addition reaction of a lower olefin (e.g., ethylene) with a saturated lower aliphatic monocarboxylic acid in the vapor phase in the presence of a heteropolyacid catalyst; the reactants are rendered substantially free of basic, nitrogen compds. by contact with an acidic, heterogeneous solid prior to being brought into contact with the heteropoly acid catalyst.

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L1 ANSWER 4 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1997119211 CAPLUS
 DOCUMENT NUMBER: 126:85811
 TITLE: Process and heteropolyacid catalysts for the preparation of esters from alkenes and carboxylic acids

INVENTOR(S): Atkins, Martin Philip; Sharma, Bhushan
 BP Chemicals Limited, UK
 Eur. Pat. Appl., 23 pp.
 CODEN: EPXXD0
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 757027	A1	19970205	EP 1996-305369	19960722
EP 757027	B1	19970205	EP 1996-305369	19960722
R: BE, DE, FR, GB, NL, SE	A1	19991124	EP 1999-113642	199960722
EP 959064	B1	20011212		
EP 959064	R: BE, DE, FR, GB, NL, SE	US 5861540	US 1996-687811	19960726
CA 2182358	AA	19970203	CA 1996-218258	19960801
JP 09118647	A2	19970506	JP 1996-203974	19960801
CN 1150385	A	19970528	CN 1996-112172	19960802
CN 1085197	B	20020522		
JP 2005298327	A2	20051027	JP 20050704950	20050713
PRIORITY APPLN. INFO.:			GB 1995-15813	A 19950802
			GB 1996-3770	A 19960222
			EP 1996-1227	A 19960625
			EP 1996-305369	A 19960722
			JP 1996-203974	A 19960801
AB Lower aliphatic esters (e.g., EtOC(=O)CH ₃) are prepared by reacting a lower olefin (e.g., H ₂ C=CH ₂) with a saturated lower aliphatic monocarboxylic acid (e.g., AcOH) in the vapor phase in the presence of a heteropolyacid catalyst, characterized in that an amount of water in the range from 1-10 mol-% (based on the total of olefin, aliphatic monocarboxylic acid and water) is added to the reaction mixture during the reaction. The presence of water enhances the ester yield. The reaction mixture may optionally contain a diether (e.g., EtoE) to minimize byproduct formation.				
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06072951	A2	19940315	JP 1993-75705	19930402
PRIORITY APPLN. INFO.:			JP 1992-180824	Al 19920708

AB The title compds. are prepared by gas-phase treating carboxylic acids with olefins in the presence of O and acid salts of heteropoly acids. A reactor containing H0.5Cs2.5PW12O40 (preparation) given was fed with a 1:13:0.02 mixed gases of acrylic acid, CH₂:CH₂, and O at 150° and atmospheric for 3 h to give 49.7% Et acylate.

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(FILE 'CAPIUS' ENTERED AT 09:10:45 ON 22 MAR 2006)

L1 FILE 'CAPIUS' ENTERED AT 09:12:50 ON 22 MAR 2006
5 S ESTER AND HETEROPOLYACID AND (GAS OR VAPOR OR VAPOUR) AND (OL
=> S ESTER AND (carrier or support or supporting or supported) AND (GAS OR VAPOR
OR VAPOUR) AND (OLEFINE OR OLEFIN) AND (CARBOXYLIC OR ACETIC OR PROPIONIC OR
ACRYLIC OR METHACRYLIC)
MISSING OPERATOR SUPPORTED) AND
The search profile that was entered contains terms or
nested terms that are not separated by a logical operator.

=> S ESTER AND (carrier or support or supporting or supported) AND (GAS OR VAPOR OR
VAPOUR) AND (OLEFINE OR OLEFIN) AND (CARBOXYLIC OR ACETIC OR PROPIONIC OR ACRYLIC
OR METHACRYLIC)

575051 ESTER

427684 ESTERS

802811 ESTER

(ESTER OR ESTERS)

264065 CARRIER

147441 CARRIERS

343441 CARRIER

(CARRIER OR CARRIERS)

441539 SUPPORT

123551 SUPPORT

524635 SUPPORT

(SUPPORT OR SUPPORTS)

89557 SUPPORTING

5 SUPPORTINGS

89561 SUPPORTING

(SUPPORTING OR SUPPORTINGS)

197847 SUPPORTED

148523 GAS

503872 GASES

1665041 GAS

(GAS OR GASES)

504059 VAPOR

70554 VAPORS

545733 VAPOR

(VAPOR OR VAPORS)

2270 VAPOUR

187 VAPOURS

2448 VAPOUR

(VAPOUR OR VAPOURS)

108 OLEFINE

250 OLEFINES

355 OLEFINE

(OLEFINE OR OLEFINES)

97518 OLEFIN

100671 OLEFINS

152638 OLEFIN

(OLEFIN OR OLEFINS)

236013 CARBOXYLIC

47 CARBOXILICS

236032 CARBOXYLIC

(CARBOXYLIC OR CARBOXYLICS)

221349 ACETIC

22 ACETICS

221358 ACETIC

(ACETIC OR ACETICS)

54670 PROPONIC

8 PROPONICS

54674 PROPONIC

(PROPONIC OR PROPONICS)

258085 ACRYLIC

1322 ACRYLICS

258457 ACRYLIC

(ACRYLIC OR ACRYLICS)

73789 METACRYLIC

6 METACRYLICS

73793 METACRYLIC

(METACRYLIC OR METHACRYLICS)

53 ESTER AND (CARRIER OR SUPPORT OR SUPPORTING OR SUPPORTED) AND

(GAS OR VAPOR OR VAPOUR) AND (OLEFINE OR OLEFIN) AND (CARBOXYLIC

OR ACETIC OR PROPONIC OR ACRYLIC OR METHACRYLIC)

=> S L2 AND (SILICOTUNGSTIC OR PHOSPHOTUNGSTIC OR SILICONOMOLYBDIC

OR SILICOVANADOTUNGSTIC OR PHOSPHOVANADOTUNGSTIC OR PHOSPHOVANADOMOLYBDIC

1331 SILICOTUNGSTIC

4744 PHOSPHOTUNGSTIC

2773 PHOSPHOMOLYBDIC

11169 SILICONOMOLYBDIC

1 SILICOVANADOTUNGSTIC

11 PHOSPHOVANADOTUNGSTIC

57 PHOSPHOVANADOMOLYBDIC

1 L2 AND (SILICOTUNGSTIC OR PHOSPHOTUNGSTIC OR PHOSPHOMOLYBDIC OR

SILICONOMOLYBDIC

1 OR PHOSPHOVANADOTUNGSTIC OR PHOSPHOVANADOMOLYBDIC)

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L3 ANSWER 1 OF 1 CAPIUS COPYRIGHT 2006 ACS on STN

AN 2004-1036764 CAPIUS

DN 142-40422

TI Support and catalyst for use in producing lower aliphatic carboxylic acid ester, catalyst manufacture process and method of use

IN Kadokawa, Etsuko; Higashi, Tomoyoshi; Oguchi, Wataru; Uchida, Hiroshi;

Narumi, Kousuke

PA Showa Denko K.K., Japan

SO U.S. Pat. Appl. Publ., 27 pp., Cont.-in-part of U.S. Ser. No. 70,259, abandoned.

CODEN: USXXCO

DT Patent

LA English

FAN.CNT 3

PATENT NO. -----

KIND -----

DATE -----

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GE, GH, GM, HR, HO, ID, IL, IN, IS, KE, KG, KR, KZ, LC, LR, LT, LU, LV, MA, MD, MG, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TM, TR, TZ, UA, UG, US, US, VN, YU, ZA, ZN, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, TZ, UW, AT, BE, CH, CY,

RW: GH, GM, KR, LS, NW, MZ, SD, SL, SZ, TZ, UW, AT, BE, CH, CY,

APPL CANTS

APPLICATION NO.	DATE
OS 2004-785229	2004-02-25
JP 2001-113624	2001-06-08
WO 2001-575532	2001-06-27
A2 20020418	
US 2004242918	A1 20041202
JP 2002079090	A2 20020319
WO 200200369	A3 20020103
WO 200200369	A2 20020418
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GE, GH, GM, HR, HO, ID, IL, IN, IS, KE, KG, KR, KZ, LC, LR, LT, LU, LV, MA, MD, MG, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TM, TR, TZ, UA, UG, US, US, VN, YU, ZA, ZN, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, TZ, UW, AT, BE, CH, CY,	

DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,
 BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG
 US 2003128371 A1 20030213 US 2001-389354 20010717
 JP 2002316046 A2 20021029 JP 2001-313675 20011207
 WO 2002054541 A1 20020822 WO 2002-JP1156 20020212
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 CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
 GM, HR, HU, ID, IL, IN, IS, KE, KG, KR, LZ, LC, LR, LS, LT,
 LU, LV, MA, MD, MG, MN, MW, NX, MZ, NO, NZ, OM, PH, PL, PT,
 RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG,
 US, UZ, VN, YU, ZA, SM, 2M, 2W, AM, AZ, BY, KG, KZ, MD, RU, TJ,
 RW: GH, GM, KE, LS, MW, MZ, SD, SL, TZ, UA, UG, ZM, ZW,
 CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR,
 BF, BJ, CF, CG, CI, CM, GA, GN, GO, GW, ML, MR, NE,
 US 2003118497 A1 20030626 US 2002-70259 20020304
 ZA 200305143 A 20030819 ZA 2002-9804 20021203
 PRAI JP 2000-192664 A 20000627 20030702
 US 2000-218803P P 20000118 PRAI JP 2000-192664 A
 JP 2001-36038 A 20010213 US 2000-218803P P
 US 2001-273343P P 20010306 JP 2000-36038 A
 WO 2001-1PP532 W 20010627 US 2001-273343P
 US 2001-889354 B2 20010717 WO 2001-1PP532
 JP 2001-873675 A 20011207 B2 W 20010627
 WO 2002-JP1156 W 20020212 JP 2001-373675
 OS CASREACT 142:40422 B2 W 20020212
 OS CASREACT 142:40422 B2 20020304

=> S L2 AND HETEROPOLYACID
 1348 HETEROPOLYACID
 936 HETEROPOLYACIDS
 1913 HETEROPOLYACID OR HETEROPOLYACIDS
 L4 1 L2 AND HETEROPOLYACID

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 L4 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN
 AN 142:40422
 DN 20041036764 CAPLUS
 TI Support and catalyst for use in producing lower aliphatic
 carboxylic acid ester, catalyst manufacture process and
 method of use
 IN Kadokawa, Etsuro; Higashi, Tomoyoshi; Oguchi, Wataru; Uchida, Hiroshi;
 Narumi, Kousuke
 PA Showa Denko K.K., Japan
 SO U.S. Pat. Appl. Publ., 27 pp., Cont.-in-part of U.S. Ser. No. 70,259,
 abandoned
 CODEN: USXXCO

DT English
 LA English
 FPN CNT 3
 PATENT NO. KIND DATE APPLICATION NO. DATE
 P1 JP 200422918 A1 20041202 US 2004-705229 20040225
 WO 2002000589 A2 20020319 JP 2001-133624 20010608
 WO 2002000589 A3 20020418 WO 2001-JP5532 20010627
 W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
 CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
 GM, HR, HU, ID, IL, IN, IS, KE, KG, KR, LZ, LC, LR, LS, LT,
 LU, LV, MA, MD, MG, MN, MW, NX, MZ, NO, NZ, PL, PT, RO, RU,
 SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN,
 YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY,
 DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,
 BJ, CF, CG, CI, CM, GA, GN, GR, ML, MR, NE, SN, TD, TG
 US 2003032437 A1 20030213 US 2001-389354 20010717
 JP 2002316048 A2 20020209 JP 2001-373675 20011207
 WO 200204541 A1 20020822 WO 2002-JP1156 20020212
 W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
 CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
 GM, HR, HU, ID, IL, IN, IS, KE, KG, KR, LZ, LC, LR, LS, LT,
 LU, LV, MR, MD, MG, MN, MW, NX, MZ, NO, NZ, PL, PT,
 RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG,
 RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ,
 CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR,
 BF, BJ, CF, CG, CI, CM, GA, GN, GO, GW, ML, MR, NE,
 US 2003118497 A1 20030626 US 2002-70259 20020304
 ZA 20030819 ZA 2002-9804 20021203
 PRAI JP 2000-192664 A 20000627 20030702
 US 2000-218803P P 20000118 PRAI JP 2000-192664 A
 JP 2001-36038 A 20010213 US 2000-218803P P
 US 2001-273343P P 20010306 JP 2000-36038 A
 WO 2001-1PP532 W 20010627 US 2001-273343P
 US 2001-889354 B2 20010717 WO 2001-1PP532
 JP 2001-373675 A 20011207 B2 W 20010627
 WO 2002-JP1156 W 20020212 JP 2001-373675
 US 2002-70259 US 2002-70259 20020304
 OS CASREACT 142:40422

=> L2 NOT L3
 L2 IS NOT A RECOGNIZED COMMAND
 The previous command name entered was not recognized by the system.
 For a list of commands available to you in the current file, enter
 "HELP COMMANDS" at an arrow prompt (>).

=> S L2 NOT L3
 L5 52 L2 NOT L3
 => LOG HOLD
 COST IN U.S. DOLLARS
 FULL ESTIMATED COST
 DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)
 CA SUBSCRIBER PRICE
 SESSION WILL BE HELD FOR 60 MINUTES
 STN INTERNATIONAL SESSION SUSPENDED AT 09:22:59 ON 22 MAR 2006
 Connecting via Winsock to STN

Welcome to STN International! Enter x:
 LOGINID:SSSPTA16232CT
 PASSWORD:
 * * * * * RECONNECTED TO STN INTERNATIONAL * * * * *
 SESSION RESUMED IN FILE 'CAPLUS' AT 09:35:56 ON 22 MAR 2006
 FILE 'CAPLUS' ENTERED AT 09:35:56 ON 22 MAR 2006
 COPYRIGHT (C) 2006 AMERICAN CHEMICAL SOCIETY (ACS)

COST IN U.S. DOLLARS SINCE FILE TOTAL
FULL ESTIMATED COST ENTRY SESSION 92.05 92.89

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) SINCE FILE TOTAL
CA SUBSCRIBER PRICE ENTRY SESSION -3.75 -3.75

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(FILE 'HOME' ENTERED AT 09:10:45 ON 22 MAR 2006)

FILE 'CAPIUS' ENTERED AT 09:12:50 ON 22 MAR 2006
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53 S ESTER AND (CARRIER OR SUPPORT OR SUPPORTING OR SUPPORTED) AND
1 S L2 AND (SILICOTUNGSTIC OR PHOSPHOTUNGSTIC OR PHOSPHOMOLYBDIC
1 S L2 AND HETEROPOLYACID
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=> D 1-52 IB1B ABS

L5 ANSWER 1 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 142:32843
DOCUMENT NUMBER: 142:32843
TITLE: Ziegler-Natta catalyst composition for ethylene
Polymerization
Campbell, Richard E., Jr.; Chen, Linfeng; Painter,
Roger B.; Reib, Robert N.; Tilston, Michael W.
Dow Global Technologies Inc., USA
PCT Int. Appl., 18 PP.

SOURCE: Coden: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO.
WO 20050421 A1 2004-US26641 20040818
W: AB, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BY, BZ, CA, CH,
CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB,
GH, GM, HR, HU, ID, IL, IS, JP, KR, KG, KP, KR, KZ, LC,
LK, LR, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM,
TJ, TM, TR, TT, TZ, UA, VC, VN, YU, ZR, ZM,
RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM,
AZ, BY, KG, K2, MD, RU, TU, TM, AT, BE, BG, CH, CY, CZ,
EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE,
SI, SK, TR, BE, BJ, CF, CG, CI, CM, GR, GN, GQ, GW,
SN, TD, TG

PRIORITY APPLN. INFO.: US 2003-50330P P 20030923
PCT Int. Appl., 35 PP.

AB A process for the polymerization of **olefin** monomers comprises the steps
of: contacting ethylene or a mixture of ethylene and one or more C4-8
olefins with a catalyst composition comprising one or more
Group 3-10 transition metal containing, Ziegler-Natta, procatalyst compds.
(e.g., MgCl₂-supported TiCl₄); one or more alkylaluminum
catalysts (e.g., triisobutylaluminum); and one or more polymerization control
agents (e.g., Et p-ethoxybenzoate). The process is characterized in that
at least one such polymerization control agent is an alkyl or aryl **ester**
of an aliphatic or aromatic (poly) **carboxylic acid** optionally containing
one or more substituents comprising a Group 13, 14, 15, or 16 heteroatom.
REFERENCE COUNT: 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 2 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2005238937 CAPIUS
DOCUMENT NUMBER: 142:316483
TITLE: Preparation of lower aliphatic **esters** by
vapor phase reaction of lower **olefins**
with lower aliphatic **carboxylic acids** in the
presence of a heteropoly acid catalyst
Fullerton, William Alan
BP Chemicals Limited, UK
PCT Int. Appl., 22 PP.
CODEN: PIXXD2
Patent

INVENTOR(S):
PATENT ASSIGNEE(S):
SOURCE:
DOCUMENT TYPE:
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO.
WO 20050317 A1 2004-GB3619 20040824
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH,
CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD,
GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, LC,
LK, LR, LS, LT, IU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA,
NO, NZ, OM, PG, PH, PT, RO, RU, SC, SD, SE, SI, SK, SY, SL, SY,
TJ, TM, TR, TT, TZ, UA, VC, VN, YU, ZR, ZM, ZW, AP,
RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM,
AZ, BY, KG, K2, MD, RU, TU, TM, AT, BE, BG, CH, CY, CZ,
EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE,
SI, SK, TR, BE, BJ, CF, CG, CI, CM, GR, GN, GQ, GW,
SN, TD, TG

PRIORITY APPLN. INFO.: GB 2003-20692 A 20030903
CARSRACT 142:316483
OTHER SOURCE(S):
AB A process for making lower aliphatic **esters** comprises reaction of
lower **olefins** with saturated lower aliphatic monocarboxylic acids in
the vapor phase using heteropoly acid catalysts at 1200-1800
KPa. In a preparation of EtOC₄ a steam comprising ethylene 23.81 g/h, HOAc
3.65 mL/h, H₂O 1 mL/h, and EtCO 0.54 mL/h at 10 barg and gas
hour space velocity of 3600 was fed to a reactor packed with silicotungstic
acid on silica at 185° to give, after 132 h on stream, byproduct
acetaldehyde 0.14 g/cat/h and MeCO 0.007 g/cat/h.
REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 3 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2004756672 CAPIUS
DOCUMENT NUMBER: 141:219428
TITLE: Processes for the production of alkenyl esters
of lower carboxylic acids and process for
the production of alkenyl alcohols
Saihata, Meiko; Uchida, Hiroshi
INVENTOR(S):
PATENT ASSIGNEE(S): Showa Denko K.K., Japan
SOURCE: PCT Int. Appl., 35 PP.
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO.
WO 2004018698 A1 20040316 20040225
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, CH, CN, CO, CR, CS,
BG, BR, BR, BN, BY, BZ, CA, CU, CZ, DE, DK, DM, DZ, EC, EC, EE, EG, ES,
ES, FI, FI, GB, GE, GH, GM, HR, HU, ID, IN,

IS, KE, KG, KP, KR, KZ, LC, LR, LS, LT, IJ, LV, MA, MD, MG, MN, MX, NA, NI, NO, NL, RW: BW, GH, GM, RS, BG, CH, CY, CZ, DE, DK, EE, ES, EL, FR, MC, NL, PT, RO, SE, SI, SK, TR, BE, BJ, GO, GW, ML, MR, NE, SN, TD, TG JP 2004339195 A2 20041202 JP 2004-63428 PRIORITY APPLN. INFO.: US 2003-45588P JP 2003-119758 A 20030424	the Periodic Table or a compound containing at least one of these elements, and palladium, wherein the outflow ratio of the compound containing alkali metal and/or alkaline earth metal is from 1.0 + 10-5 to 0.01V/h. REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT	IS, KE, KG, KP, KR, KZ, LC, LR, LS, LT, IJ, LV, MA, MD, MG, MN, MX, NA, NI, NO, NL, RW: BW, GH, GM, RS, BG, CH, CY, CZ, DE, DK, EE, ES, EL, FR, MC, NL, PT, RO, SE, SI, SK, TR, BE, BJ, GO, GW, ML, MR, NE, SN, TD, TG JP 2004339195 A2 20041202 JP 2004-63428 PRIORITY APPLN. INFO.: US 2003-45588P JP 2003-119758 A 20030424	the Periodic Table or a compound containing at least one of these elements, and palladium, wherein the outflow ratio of the compound containing alkali metal and/or alkaline earth metal is from 1.0 + 10-5 to 0.01V/h. REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT
AB A process for producing a lower aliphatic carboxylic acid alkanyl, comprising reacting lower olefin, lower aliphatic carboxylic acid and oxygen in a gas phase in the presence of a catalyst comprising a support having supported thereon a catalyst component containing a compound containing alkali metal and/or alkaline earth metal, an element belonging to Group 11 of the Periodic Table or a compound containing at least one of these elements, and palladium, wherein the conversion of the lower aliphatic carboxylic acid is 80% or less or the concentration of the lower aliphatic carboxylic acid in the reactor outlet is 0.5 mol% or more. REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT	15 ANSWER 4 OF 52 CARPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 2004:156610 CARPLUS DOCUMENT NUMBER: 141:279426 TITLE: Production processes of lower aliphatic carboxylic acid alkanyl esters and alkanyl alcohol INVENTOR(S): Saito, Meiko; Uchida, Hiroshi SOURCE: Showa Denko K.K., Japan PCT Int. Appl., 36 pp. Coden: PIXBD2 Patent DOCUMENT TYPE: LANGUAGE: English FAMILY ACC. NUM. COUNT: 1 PATENT INFORMATION:	AB A process for producing a lower aliphatic carboxylic acid alkanyl, comprising reacting lower olefin, lower aliphatic carboxylic acid and oxygen in a gas phase in the presence of a catalyst comprising a support having supported thereon a catalyst component containing a compound containing alkali metal and/or alkaline earth metal, an element belonging to Group 11 of the Periodic Table or a compound containing at least one of these elements, and palladium, wherein the conversion of the lower aliphatic carboxylic acid is 80% or less or the concentration of the lower aliphatic carboxylic acid in the reactor outlet is 0.5 mol% or more. REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT	15 ANSWER 4 OF 52 CARPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 2004:156610 CARPLUS DOCUMENT NUMBER: 141:279426 TITLE: Production processes of lower aliphatic carboxylic acid alkanyl esters and alkanyl alcohol INVENTOR(S): Saito, Meiko; Uchida, Hiroshi SOURCE: Showa Denko K.K., Japan PCT Int. Appl., 36 pp. Coden: PIXBD2 Patent DOCUMENT TYPE: LANGUAGE: English FAMILY ACC. NUM. COUNT: 1 PATENT INFORMATION:
WO 2004078696 A1 20040916 WO 2004-0225 W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DL, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, ID, IL, IR, IS, KE, KG, KP, KR, LZ, MC, MN, MX, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, NA, NI, NO RW: BW, GH, GM, KE, LS, MW, SD, SL, SZ, TZ, WG, ZM, BG, CH, CY, CZ, DE, DK, EE, ES, FL, FR, GR, HU, IE, IR, LU, MC, NL, PT, RO, SE, SI, SK, TR, BE, BJ, CE, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG EP 1603859 A1 20051214 EP 2004-714478 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, NC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, C2, EE, HU, SK JP 2004339194 A2 20041202 JP 2004-63363 JP 2003-61494 A 20040308 US 2003-453951P P 20030313 JP 2003-119757 A 20030424 WO 2004-J2214 W 20040225	15 ANSWER 5 OF 52 CARPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 2003:333092 CARPLUS DOCUMENT NUMBER: 138:304650 TITLE: High activity silica gel carrier catalyst composition for ethylene polymerization or copolymerization INVENTOR(S): Luo, Ruiqo; Gao, Kejing; Zhao, Qinfang; An, Jinyuan; Yang, Hua; Huo, Jinsheng; PATENT ASSIGNEE(S): China Petrochemical Corp.; Peoples Rep. China; Beijing Research Institute of Chemical Industry; SINOPEC SOURCE: Faming Zhanli Shengqing Gongtai Shuomingshu, 16 pp. DOCUMENT TYPE: LANGUAGE: Chinese FAMILY ACC. NUM. COUNT: 1 PATENT INFORMATION:	15 ANSWER 5 OF 52 CARPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 2003:333092 CARPLUS DOCUMENT NUMBER: 138:304650 TITLE: High activity silica gel carrier catalyst composition for ethylene polymerization or copolymerization INVENTOR(S): Luo, Ruiqo; Gao, Kejing; Zhao, Qinfang; An, Jinyuan; Yang, Hua; Huo, Jinsheng; PATENT ASSIGNEE(S): China Petrochemical Corp.; Peoples Rep. China; Beijing Research Institute of Chemical Industry; SINOPEC SOURCE: Faming Zhanli Shengqing Gongtai Shuomingshu, 16 pp. DOCUMENT TYPE: LANGUAGE: Chinese FAMILY ACC. NUM. COUNT: 1 PATENT INFORMATION:	15 ANSWER 6 OF 52 CARPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 2003:333092 CARPLUS DOCUMENT NUMBER: 138:304650 TITLE: High activity silica gel carrier catalyst composition for ethylene polymerization or copolymerization INVENTOR(S): Luo, Ruiqo; Gao, Kejing; Zhao, Qinfang; An, Jinyuan; Yang, Hua; Huo, Jinsheng; PATENT ASSIGNEE(S): China Petrochemical Corp.; Peoples Rep. China; Beijing Research Institute of Chemical Industry; SINOPEC SOURCE: Faming Zhanli Shengqing Gongtai Shuomingshu, 16 pp. DOCUMENT TYPE: LANGUAGE: Chinese FAMILY ACC. NUM. COUNT: 1 PATENT INFORMATION:
AB A process for producing a lower aliphatic carboxylic acid alkanyl, comprising reacting lower olefin, a lower aliphatic carboxylic acid and oxygen in a gas phase in the presence of a catalyst comprising a support having supported thereon a catalyst component containing a compound containing alkali metal and/or alkaline earth metal, an element belonging to Group 11 of	WO 200202638 A1 20020404 W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CO, CR, CU, CZ, DE, DK, DM, DZ, ES, FI, GB, GD, GE, GH, GR, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, LZ, MC, MN, MW, MX, NZ, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UP, UG, UZ JP 2003-119757 A 20030424 WO 2004-J2214 W 20040225 CN 2000-123560 20000822 WO 2001-CN1264 20010822 CN 1339509 A 20020313 WO 200202638 A1 20020404 W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CO, CR, CU, CZ, DE, DK, DM, DZ, ES, FI, GB, GD, GE, GH, GR, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, LZ, MC, MN, MW, MX, NZ, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UP, UG, UZ	WO 200202638 A1 20020404 W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CO, CR, CU, CZ, DE, DK, DM, DZ, ES, FI, GB, GD, GE, GH, GR, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, LZ, MC, MN, MW, MX, NZ, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UP, UG, UZ	WO 200202638 A1 20020404 W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CO, CR, CU, CZ, DE, DK, DM, DZ, ES, FI, GB, GD, GE, GH, GR, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, LZ, MC, MN, MW, MX, NZ, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UP, UG, UZ

PRIORITY APPLN. INFO. :		CN 2000-123560	A	200008222000
OTHER SOURCE(S) :	MARPAT 138:304690	WO 2001-CN1264	W	200108222000
AB	The catalyst component contains a Ti-containing active component carrier, where the Ti-containing active component is a porous inert carrier, where the Ti-containing active component is a reaction product of at least one type of Ti compound (such as TiCl ₄) with at least one type of Mg compound (such as MgCl ₂) and at least one type of reaction donor compound (such as Et acetate) in a molar ratio of 1:0.5-50, 50, and may also contain at least one type of halide modifier (such as Cl ₃ CCl ₂ OH) (at a molar ratio of Ti compound/halide modifier = 1.0-5-50), and the porous inert carrier is spherical or spherical-like silica gel having average particle size 10-100 μm and surface area 300-1000 m ² /g. The catalyst component is combined with a tri-alkyl aluminum compound (such as tri- <i>t</i> -butyl aluminum) and used in gaseous slurry polymerization or copolymer of ethylene, and especially used in preparing high-quality LDPE resin by gaseous fluidized bed condensation technique.			
15. ANSWER 7 OF 52	CAPLUS COPYRIGHT 2006 ACS on STN			
ACCESSION NUMBER:	2002738941 CAPLUS			
DOCUMENT NUMBER:	137149510			
TITLE:	Preparation of a saturated carboxylic acid ester and use of water-soluble palladium complex			
INVENTOR(S) :	Seayad, Jayasree; Sseyad, Abdul Majed; Sarkar, Bipan Ranjan; Chaudhari, Raghunath Vilthal			
PATENT ASSIGNEE(S) :	Council of Scientific & Industrial Research, India			
SOURCE:	U.S. Pat. Appl. Publ., 9 pp.			
CODEN: USXXCO				
Patent				
LANGUAGE:	English			
FAMILY ACC. NUM. COUNT:	1			
PATENT INFORMATION:				
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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US 2002131964	A1	20020926	US 2001-113086	20010320
US 6479693	B2	20021112	US 2001-813086	20010320
PRIORITY APPLN. INFO. :	MARPAT 137:249510			
OTHER SOURCE(S) :	AB A carboxylic acid ester R ₃ R'CC(=O)R ₂ CO ₂ R' (I) is prepared where R = alkyl or aryl, R ₁ = aryl, substituted aryl, naphth-substituted naphthyl or alkyl, R ₂₋₅ = H or alkyl, by reacting an olefin R3C:CR1H ₂ (R ₁ = aryl, substituted aryl, naphthyl or substituted naphthyl or alkyl, R ₂₋₄ = H or alkyl), in the presence of alc. and an organic solvent and supported aqueous phase Pd complex catalyst, and in presence or absence of a protonic acid and an alkali metal halide, under CO atmospheric, cooling the reaction mixture to am temperature, depressurizing the reactor, flushing the reaction vessel with inert gas, separating the catalyst by filtration, and removing the solvent and isolating I. Thus, styrene (0.144 mol), MeOH, cyclohexane, and Pd(RTPPS) ₃ [RTPPS = tri-(sodium 3-(2-sulfonatoethyl)phosphinyl)] was heated 75 °C, pressurized to 500 psig with CO, and stirred for 12 h to give a mixture of 2- and 3-methylphenyl propionates, selectivity of 34.26% and 4			

ACCESSION NUMBER:	2002-637632 CAPLUS			
DOCUMENT NUMBER:	137:17135			
TITLE:	Process for the use of and supports for the manufacture of catalysts for producing lower aliphatic carboxylic acid esters via the addition-esterification reaction of lower aliphatic carboxylic acids with lower alkenes			
INVENTOR(S):	Kadowaki, Etsuko; Narumi, Kousuke; Uchida, Hiroshi			
PATENT ASSIGNEE (S):	Showa Denko K. K., Japan			
SOURCE:	PCT Int. Appl., 45 pp.			
DOCUMENT TYPE:	Patent			
LANGUAGE:	English			
FAMILY ACC. NUM. COUNT:	3			
PATENT INFORMATION:				
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2002064541 W	A1	20020822	WO 2002-JP1156	20020212
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, ET, GB, GD, GH, GM, HR, ID, IL, IN, IS, KE, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MR, MD, MG, MN, MK, MW, MX, MY, NO, NZ, OM, PH, PL, PT, RO, RU, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, 2M, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IL, LU, MG, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, MI, MR, NE, SD, TD, JP 2002316948 A2	20021029	JP 2001-373675	20011207	
EP 1360166 A1	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR	20031112	EP 2002-711471	20020212
BR 200207225 A	20040309	BR 2002-7225	20020212	
CN 1491204 A	20040421	CN 2002-804859	20020212	
US 200318497 A1	20030826	US 2002-70259	20020304	
ZA 200305143 A	20040312	ZA 2003-5143	20030718	
US 2004242918 A1	20041202	US 2004-783229	20040225	
PRIORITY APPLN. INFO.:				
AB	A siliceous support for use in a catalyst for producing a lower aliphatic carboxylic acid esters (e.g., Et acetate) by the esterification-addition reaction of a lower olefin (e.g., ethylene) with a lower aliphatic carboxylic acid (e.g., acetic acid) in the gas phase is described and the support has a silicon content of 39.7-46.31 by mass or a silicon content of >30 N; the catalyst is a heteropoly acid or a crush support. By the use of a catalyst comprising the support, a lower aliphatic carboxylic acid ester is produced from a lower olefin and a lower aliphatic carboxylic acid without causing a great reduction of catalytic activity or cracking or abrasion of the catalyst.	US 2001-36038	A 20010213	
	US 2001-273343 P	P 20010306		
	JP 2001-373675	A 20011207		
	JP 2000-192864	A 20000627		
	US 2000-218803 P	P 20000718		
	WO 2001-JP5532	W 20010627		
	US 2001-889354	B2 20010717		
	WO 2002-JP1156	W 20020212		
	US 2002-70259	B2 20020304		
REFERENCE COUNT:	4			
RECORD:	THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT			

TITLE: Process for producing ethylene-**olefin**
INVENTOR(S): Ford, Randal Ray; Vanderbilt, Jeffrey James; Williams, Darryl Stephen
PATENTEE(S): Eastman Chemical Co., USA
SOURCE: PCT Int. App., 64 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2000075202	A1	20001214	WO 1999-US22957	19991014
W: BR, CA, CN, JP, MX RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE	AA	20001214	US 1999-386545	19990831
US 6417301	BI	20020709	US 1999-386545	19991014
CA 2375621	AA	20001214	CA 1999-2375621	19991014
BR 9917354	A	20020226	BR 1999-137354	19991014
EP 1183284	A1	20020306	EP 1999-931735	19991014
EP 1183284	BI	20050427	EP 1999-US22957	19991014
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI	T2	20030114	JP 2001-502482	19991014
JP 2003501526	PRIORITY APPLN. INFO. :	US 1999-131979P	P 19990607	AB
US 1999-36545	US 1999-36545	A 19990831	AB	
WO 1999-US22957	WO 1999-US22957	W 19991014	AB	

AB The process uses Ziegler-Natta catalysts for producing ethylene-**olefin** interpolymers having a given melt index and d, and a reduced melting peak temperature (T_m). The process involves contacting and ≥ 1 other **olefin** under polymerization conditions with a Ziegler-Natta catalyst and ≥ 1 modifier comprising ≥ 1 element from Group 15 and/or Group 16 in amounts sufficient to reduce the melting peak temperature of the interpolymers.

REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

15 ANSWER 10 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1999:168033 CAPIUS
DOCUMENT NUMBER: 131:118312
TITLE: Attrition resistant catalysts and sorbents based on heavy metal poisoned FCC catalysts

INVENTOR(S): Gangwal, Santosh; Jothimurugesan, Kandaswamy
PATENT ASSIGNEE(S): Research Triangle Institute, USA
SOURCE: U.S., 9 pp.
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5928980	A	19990727	US 1997-793669	19970206

PRIORITY APPLN. INFO. : AB A heavy metal poisoned, spent FCC catalyst with a new catalytic metal or metal salt to provide an attrition resistant catalyst or sorbent for a different synthesis, and sorbents for removal of sulfur **gases** from fuel **gases** and fine **gases**. The heavy metal contaminated FCC catalyst is directly used as a support for preparing catalysts

having new catalytic properties and sorbents having new sorbent properties, without removing or passivating the heavy metals on the spent FCC catalyst as an intermediate step.

REFERENCE COUNT: 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 11 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1998:76040 CAPIUS
DOCUMENT NUMBER: 130:1188
TITLE: A process for the preparation of a magnesium halide-supported metallocene polymerization catalyst

INVENTOR(S): Sengartha, Sivaram; Srinivasan, Swaminathan
PATENTEE(S): Council of Scientific and Industrial Research, India
SOURCE: Eur. Pat. Appl., 10 pp.
CODEN: EPXXDM

DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 87484	A1	19981118	EP 1997-303286	19970514
EP 87484	B1	20041215	R: BE, CH, DE, FR, GB, LI	IN 1997-DE795

PRIORITY APPLN. INFO. : AB A magnesium halide-supported metallocene catalyst, useful for polymerization of **olefins** in slurry or **gas** phase, is manufactured by (a) preparing a solution (solution A) of the magnesium halide compound via Grignard decomposition which consists of preparing the slurry of magnesium metal in an electron donor solvent, and heating the slurry of magnesium metal to 0°C to 50°C for a period of 10 min to 4 h while adding a dihaloalkane compound; (b) sep. preparing a solution of metallocene compound into the same electron donor solvent (solution B); (c) heating the solution B to 0°C to 50°C for a period ranging between 10 min to 1 h; (d) adding solution B into solution A in a period ranging between 10 min to 2 h, while keeping the temperature in the range of 0 to 50°C; (e) cooling the resultant homogeneous solution to room temperature under inert atmospheric;

(f) pouring the reaction mixture into a liquid hydrocarbon medium in which all the components will be partly or completely insol. to precipitate the catalyst; (g) separating the solid catalyst precipitated by conventional methods; (h) washing the solid catalyst by a hydrocarbon solvent; and (i) drying the solid under vacuum at a temperature ranging between 0 to 50°C.

REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 12 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1995:44065 CAPIUS
DOCUMENT NUMBER: 122:188400

TITLE: Catalyst and its use in the preparation of unsaturated carboxylic acid esters

INVENTOR(S): White, James F.; Slawski, Barbara; White, Geoffrey
PATENTEE(S): Engelhard Corp., USA
SOURCE: U.S., 11 pp.
CODEN: USXXAM

DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 1997-793669	US 1997-793669	19970206	AB	

PRIORITY APPLN. INFO. : AB Imregnating the poisoned catalyst with a new catalytic metal or metal salt to provide an attrition resistant catalyst or sorbent for a different synthesis, and sorbents for removal of sulfur **gases** from fuel **gases** and fine **gases**. The heavy metal contaminated FCC catalyst is directly used as a support for preparing catalysts

PRIORITY APPLN. INFO.: -----

AB Title catalysts comprise (i) ≥ 1 palladium group metals and/or compounds thereof; (ii) gold and/or compounds thereof; and (iii) optionally, ≥ 1 promoters selected from Cu, Ni, Co, Fe, Mn, Pb, Ag and their compounds. Preferably these catalyst compns. contain ≥ 1 promoters wherein the gram ratio of metal in the promoter to Palladium group metal is ≤ 0.4 , and the catalysts are deposited on a support material treated with ≥ 1 alkali metal bases and ≥ 1 alkali metal silicates prior to deposition. In some cases, the catalysts are further impregnated with ≥ 1 alkali or alkaline-earth carboxylates as promoters and activators. Ethylenically unsat'd esters are prepared with improved activity and selectivity by reacting in the vapor phase ≥ 1 olefinic compds. with ≥ 1 carboxylic acid and mol. oxygen in the presence of the foregoing catalyst compns. Thus, vapor-phase reaction of ethylene at 140° with HOAc and O over silica beads that were pretreated with KOH and K silicate solution at 16.2 and 127.2 g/L beads, resp., and impregnated with Na₂PdCl₄, NaAuCl₄, CuCl₂, and KOC to contain 4.1, 1.8, and 22 g./L catalyst of Pd, Au, Cu, and KOC, resp., gave vinyl acetate at space-time yield 158.3 (95% selectivity) and selectivity 96.3% (125 space-time yield).

15 ANSWER 13 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1994-056532 CAPLUS
DOCUMENT NUMBER: 121-265552 CAPLUS
TITLE: Components and catalysts for the Polymerization of Olefins.
INVENTOR(S): Sacchetti, Mario; Cuffiani, Illaro; Pennini, Gianni
SOURCE: Spherilene S.r.l., Italy
DOCUMENT TYPE: Eur. Pat. Appl., 15 pp.
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION: -----

PATENT NO. DATE KIND APPLICATION NO. DATE
EP 601525 A1 19940115 EP 1993-19646 19931206
EP 601525 B1 19990210 IN 1993-C121 19931204
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, NL, PT, SE
IN 181561 A 19980111 AT 1993-19646 19931206
AT 176676 E 19990215 ES 1993-19646 19931206
ES 2129063 T3 19990215 19931206
AU 9332295 A1 19940223 AU 1993-52295 19931208
AU 675551 B2 19961114 19931208
ZA 9309210 A 19940808 ZA 1993-9210 19931208
JP 07300507 A2 19951114 JP 1993-38020 19931208
RU 2126420 C1 19990220 RU 1993-54128 19931208
NO 9304496 A 19940613 NO 1993-496 19931209
IL 107958 A1 19980324 IL 1993-107958 19931209

PRIORITY APPLN. INFO.: -----

AB Title catalysts comprise (i) ≥ 1 palladium group metals and/or compounds thereof; (ii) gold and/or compounds thereof; and (iii) optionally, ≥ 1 promoters selected from Cu, Ni, Co, Fe, Mn, Pb, Ag and their compounds. Preferably these catalyst compns. contain ≥ 1 promoters wherein the gram ratio of metal in the promoter to Palladium group metal is ≤ 0.4 , and the catalysts are deposited on a support material treated with ≥ 1 alkali metal bases and ≥ 1 alkali metal silicates prior to deposition. In some cases, the catalysts are further impregnated with ≥ 1 alkali or alkaline-earth carboxylates as promoters and activators. Ethylenically unsat'd esters are prepared with improved activity and selectivity by reacting in the vapor phase ≥ 1 olefinic compds. with ≥ 1 carboxylic acid and mol. oxygen in the presence of the foregoing catalyst compns. Thus, vapor-phase reaction of ethylene at 140° with HOAc and O over silica beads that were pretreated with KOH and K silicate solution at 16.2 and 127.2 g/L beads, resp., and impregnated with Na₂PdCl₄, NaAuCl₄, CuCl₂, and KOC to contain 4.1, 1.8, and 22 g./L catalyst of Pd, Au, Cu, and KOC, resp., gave vinyl acetate at space-time yield 158.3 (95% selectivity) and selectivity 96.3% (125 space-time yield).

15 ANSWER 14 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
ACCESSION NUMBER: 1994-056532 CAPLUS
DOCUMENT NUMBER: 120-322766 CAPLUS
TITLE: Preparation of unsaturated carboxylic acid esters with palladium catalysts
INVENTOR(S): Harada, Hiromi; Hamachi, Hideyuki; Nishizuka, Masaya; Kato, Takao; Saito, Toshihiro
PATENT ASSIGNEE(S): Tosoh Corp., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
CODEN: JRXKAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION: -----

PATENT NO. KIND DATE APPLICATION NO. DATE
----- A2 19940201 -----
JP 06023082 ----- JP 1992-200102 19920706
PRIORITY APPLN. INFO.: -----
OTHER SOURCE(S): CARSRACT 120-322766
AB Unst. carboxylic acid esters are prepared by reaction of olefins, O, and AcOH in gas phases in presence of Pd catalysts prepared by ion exchange of OH on the surfaces of catalyst supports with Pd ammine complexes, optional calcination under O-containing gases, reduction of the supports, and supporting alkali metal acetates on the supports. Silica was impregnated with an aqueous NH₃ solution of [Pd(NH₃)₄C12], dried at 110° for 3 h, heated at 400° for 5 h, reduced by H at 300° for 5 h, impregnated with aqueous AcOH, and dried at 110° for 3 h to prepare a Pd- and AcOH-containing catalyst. A 50:9:7:33 mixture of propylene, O, AcOH, and N was passed through the catalyst at 150°, 5 atm, and 13.6 NL/h to give 307 g./h allyl acetate with 95.8%

selectivity.

L5 ANSWER 15 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1994:191137 CAPIUS
DOCUMENT NUMBER: 120:191137
TITLE: Preparation of lower fatty acid **esters**
INVENTOR(S): Suzuki, Toshiro; Takahashi, Shunsei; Sano, Kenichi
PATENT ASSIGNEE(S): Showa Denko Kk, Japan
SOURCE: Jpn. Kokai Tokyo Koho, 4 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
PRIORITY APPLN. INFO.: JP 03294894 A2 19931109 JP 1992-101364 DATE 19920421
IN 2850636 B2 199012 IN 2000-D6957 20001025
IN 189050 A 20021214 JP 1992-101364 A 19920421

PRIORITY APPLN. INFO.: CASREACT 120:191137
OTHER SOURCE(S): AB The title compds. are prepared by **gas-phase esterification of** lower fatty acids with lower **olefins** using **supported**

catalysts of heteropoly acid salts of Li, Cu, Mg, and/or Ga. A reactor

containing Li phosphotungstate supported on SiO₂ (preparation given) was

fed with mixed **gases** containing AcOH and CH₂:CH₂ at 150° and

5 kg/cm²-gage to give 91.8% Et acetate.

L5 ANSWER 16 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:133032 CAPIUS
DOCUMENT NUMBER: 117:33032
TITLE: Steam stripping for removal of organic pollutants from water. 2. **vapor-liquid equilibrium**
AUTHOR(S): Huang, Yng Long; Olson, James D.; Keller, George E., II
CORPORATE SOURCE: Union Carbide Chem. and Plast. Co. Inc., South
Charleston, WV, 25303, USA
INDUSTRIAL & ENGINEERING CHEMISTRY RESEARCH (1992),
31(7), 1759-68
CODEN: IECRED; ISSN: 0888-5885
JOURNAL

DOCUMENT TYPE: English
LANGUAGE:

AB In support of the simple tools for steam stripping presented in part 1, the estimation of the infinite-dilution vapor-liquid equilibrium constants (K_{∞}) for organic pollutants in dilute aqueous soils are discussed. A database of K_{∞} at 1 atm and 100° as well as at 25° was established for a number of common organic pollutants. In addition, a correlation of K_{∞} with mol. structure was formulated. The concept that the effectiveness of stripping an organic pollutant from water is mainly determined by its hydrophobicity (indicated by its low aqueous solubility) rather than by its pure-component volatility is also clarified. On the basis of the guideline proposed in Part 1, the estimated K_{∞} values reveal that steam stripping is effective in removing hydrophobic (sparingly soluble) pollutants such as paraffins, olefins, aroms, halogenated hydrocarbons, and compds. with dominant hydrocarbon segments.

L5 ANSWER 17 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:237786 CAPIUS
DOCUMENT NUMBER: 116:237786
TITLE: Hollow cylindrical carrier as catalyst support for production of unsaturated esters

INVENTOR(S): Matsumoto, Kenji; Fuchigami, Yoshiro
PATENT ASSIGNEE(S): Kuraray Co., Ltd, Japan
SOURCE: Eur. Pat. Appl., 16 pp.
DOCUMENT TYPE: CODEN: EPXXDW
Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
PATENT NO. KIND DATE APPLICATION NO. DATE
EP 464633 A1 19920108 EP 1991-110562
EP 464633 R: BE, DE, FR, GB, IT 19940119
JP 04227069 A2 19920817 JP 1991-99591
US 5371277 A 19941206 JP 1990-1717125
JP 1990-1717125 A 19900703
US 1991-5233376 B1 19910628
US 1993-52292 B1 19930426
AB Production of unsatd. **esters** with high catalyst activity and low pressure drop in the catalyst layer is achieved by **gas-phase** reaction of **olefin**, **carboxylic acid**, and O in the presence of a catalyst (e.g. Pd) **supported** on a hollow cylindrical carrier having ≥ 1 through channel. A **gas** containing C₂H₄ 72%, O₂ 8%, and AcOH 20 volume% was passed through a catalyst layer (containing Pd 5.0, Au 0.5, and KOAc 20 g/L on a hollow cylindrical silica) at 160° and 2000 h⁻¹ giving vinyl acetate with selectivity 94% and pressure drop 0.20 kg/cm², vs. 95 and 0.26 resp., when using a spherical silica **support**.

L5 ANSWER 18 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:227394 CAPIUS
DOCUMENT NUMBER: 116:227394
TITLE: Chromatographic method for analysis of aliphatic olefin alcohols, acetates and ketones

INVENTOR(S): Nestrova, I. P.; Rastegieva, V. M.
CORPORATE SOURCE: All-Union Scientific-Research Institute of Biological Methods of Plant Protection, USSR
SOURCE: U.S.S.R. From: Otkrytiya, Izobret. 1991, (31), 182.
DOCUMENT TYPE: Patent
LANGUAGE: Russian
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
PATENT NO. KIND DATE APPLICATION NO. DATE
SU 1672352 A1 19910823 SU 1988-4445933 19880513
SU 1672352 A1 19910823 SU 1988-4445933 19880513
AB The mixture is separated in a capillary column with a stationary phase in a carrier-gas flow with subsequent detection of separated isomers. To increase separation selectivity and expand the circle of sample substances - among them insect sex attractants, liquid-crystal cholesteryl-p-methoxybenzoate is used as the stationary phase, and the separation is performed at 170-190° for a carrier-gas rate in the column over the interval of 6.0-15.0 cm/s.

L5 ANSWER 19 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:173577 CAPIUS
DOCUMENT NUMBER: 116:173577
TITLE: Preparation of catalysts for unsaturated ester manufacture

INVENTOR(S): Ikumi, Toshi; Takagi, Isato; Morofuji, Masamitsu; Fujiwara, Hideetsu; Yoshida, Soichi
Tosoh Corp., Japan Synthetic Rubber Co., Ltd.
PATENT ASSIGNEE(S):

SOURCE:	Jpn. Kokai Tokkyo Koho, 8 pp		
DOCUMENT TYPE:	Patent		
LANGUAGE:	Japanese		
FAMILY ACC. NUM. COUNT:	1		
PATENT INFORMATION:	PATENT NO.	KIND	DATE
	-----	-----	-----
JP 03275141	A2	19911205	JP 1990-7713577
PRORITY APPLN. INFO.:	OTHER SOURCE(S):	CASREACT 116:173577	
AB	Pd catalysts for preparation of unsaturated esters carbonylic acids, and O are prepared by treating supports in aqueous tartaric acid containing O-1,1-diaacetoxysubstituted reduction. A mixture of PdCl ₂ 13.4, Sr dissolved in an aqueous solution containing 25% then 150		
24	9	silica gel was stirred in the solution, dried 20 h, and reduced in H at 200° for 2 h and at 400 The solid was then impregnated in an aqueous soil	
24	9	CsOAc and dried to give a Pd catalyst. A mixed catalyst at 185° for 100 h to give 95.3% diaetoxycetyl, AcOH, O, and N (20/20/12/4) was diaetoxycetyl, 1,4-isomer.	
L5	ANSWER 20 OF 52	CAPLUS	COPYRIGHT 2006 ACS on ST
	ACCESSION NUMBER:	1991-0444 CAPLUS	
	DOCUMENT NUMBER:	115:90244	
	TITLE:	Preparation and uses Ziegler catalysts	
	INVENTOR(S):	Bailly, Jean Claude Andre; Baud, BP Chemicals Ltd., UK; BP Chem	
	PATENT ASSIGNEE(S):	Eur. Pat. Appl., 11 pp.	
	SOURCE:	CODEN: EFXXDW	
	DOCUMENT TYPE:	Patent	
	LANGUAGE:	English	
	FAMILY ACC. NUM. COUNT:	1	
PATENT INFORMATION:	PATENT NO.	KIND	DATE
	-----	-----	-----
EP 437080	A1	19910717	EP 1990-311990
FR R. AT, BE, CH, DE, FR, GB, GR, IT, I	A1	19910717	EP 1990-311990
FR 26566615	A1	19930507	FR 1990-651990
US 5106805	B1	19920421	US 1990-661990
AU 9068355	A	19910705	AU 1990-661990
CA 2033028	A1	19910705	CA 1990-711990
NO 0005634	AA	19910705	NO 1990-541990
JP 04039309	A2	19920210	JP 1990-411990
FI 9100020	A	19910705	FI 1991-211991
HU 59102	A2	19920629	HU 1991-111991
CN 1033243	A	19910724	CN 1991-111991
BR 9100019	A	19911022	BR 1991-111991
PRIORITY APPLN. INFO.:	-----	-----	FR 1990-111990
AB	Ziegler-Natta catalysts, useful for the polymerization the manufacture of elastomers, are comprising by cohydrocarbon as spheroidal support comprising MgC ₂ electron donor compound free of labile H, successively donor compound containing labile H, and then with capable of reducing V and Ti compds., washing the hydrocarbon, and then contacting the washed spherical hydrocarbon-soluble V and Ti compds. Thus, spherical		

support (prepared by reacting Bu ₂ Bg with tert BuCl and diisoamyl ether) in C6H ₆ was heated with C6H ₆ containing BuOH, decanted, and then successively contacted with Et ₃ Al, VOCl ₃ and TiCl ₄ to give as spherical catalyst having the following molar ratio: V/Mg = 0.15, Ti/Mg = 0.04, Al/Mg = 0.15, and Cl/Mg = 2.6. The catalyst was used for the gas -phase copolymer of ethylene and propylene.									
15	ANSWER 21 OF 52	CAPLUS	COPYRIGHT 2006 ACS on STN						
ACCESSION NUMBER:	1991:187321	CAPLUS							
DOCUMENT NUMBER:	11:187321								
TITLE:	Preparation of elastomeric propylene copolymers by gas phase polymerization in presence of Ziegler-Natta catalysts								
INVENTOR(S) :	Bailly, Jean Claude Andre; Bordere, Louis								
PATENT ASSIGNEE(S) :	BP Chemicals Ltd., UK								
SOURCE:	Eur. Pat. Appl., 14 pp.								
DOCUMENT TYPE:	Patent								
LANGUAGE:	English								
FAMILY ACC. NUM. COUNT:	1								
PATENT INFORMATION:									
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE					
EP 415588	A1	19910306	EP 1990-308836	-----	19900810				
EP 415588 R:	B1	19901119							
FR 2651234 AT:	DE, CH, DK, ES, GB, GR, IT, LI, LU, NL, SE	19910301	FR 1999-11523	-----	19900829				
FR 2651234 FR:	B1	19903012							
AT 100475	E	19900215	AT 1990-308836	-----	19900810				
ES 2062387	T3	19901216	ES 1990-308836	-----	19900810				
ES 2062387 IN:	A	19901213	IN 1990-DE820	-----	19900816				
CA 203653 AA:	AA	19910301	CA 1990-023553	-----	19900820				
CA 203653 AU:	A1	19910307	AU 1990-01330	-----	19900824				
AU 622573 B2:	B2	199020409	DD 1990-343664	-----	19900827				
DD 29936 A5:	A5	199020319	DD 1990-343664	-----	19900827				
NO 900763 NO:	A	19910301	NO 1990-3763	-----	19900828				
NO 175263 B:	B	19900613							
NO 175263 C:	C	19900921							
HU 54715 A2:	A2	19910328	HU 1990-5509	-----	19900828				
JP 03093803 A2:	A2	19910416	JP 1990-224507	-----	19900828				
BR 9004245 A:	A	19910903	BR 1990-1245	-----	19900828				
BR 9004245 CN:	A	19910327	CN 1990-107007	-----	19900829				
CN 1050195 A:	A	19910327	FR 1999-11523	A 19800829					
PRIORITY APPLN. INFO.:			EP 1990-308836	A 19900810					
AB	Elastomeric copolymers of 20-80% propylene (I) with 80-20% ethylene (II) and/or 1-butene (III) and optionally, >1 C5-12-olefins are prepared by a 2-stage procedure, prepolymg. in the first stage I, optionally mixed with II and/or C4-12- α -olefin, in the presence of a Ziegler-Natta catalyst and a cocatalyst consisting of a trialkylaluminum halide not complexed with an external electron donor compound, followed by a gas-phase copolymer of the prepolymer with a mixture of I, II, and/or III and optionally a C5-12- α -olefin in a proportion so that I represents 20-80 volume% of the copolymerizable olefins. The Ziegler-Natta catalyst is prepared by contacting MgCl ₂ support with an internal electron donor compound, e.g., aromatic carboxylic acid esters, and then with TiCl ₄ , provided the support is initially presoaked by the different organic electron donor compound which is free from labile H, and provided the support consists of 80-99.5 mol% MgCl ₂ and 0.5-20 mol% electron donor compound. Thus, a preexisting support comprises MgCl ₂ in hexane (IV) was decanting, washing, and resuspension in IV to give a particulate catalyst containing 6.5% titations per Mg atom and having a specific surface area of 100-200 m ² /g. The catalyst was used for the gas -phase copolymer of ethylene and propylene.								

size distribution. I was then prepolynd. in the presence of the catalyst and Et₂AlCl in IV to give a prepolymer suspension in IV. The prepolymer was then copolyd. in a fluidized bed reactor with I and II in the presence of anhydrous II-I copolymer and (iso-Bu)₃Al to give an agglomerated II-I copolymer containing 63% II and having a bulk d. 0.45 g/cm³.

15. ANSWER 22 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1985:023053

DOCUMENT NUMBER: 103:2053

TITLE: Deodorization of polyolefins

SOURCE: Ube Industries, Ltd., Japan

CODEN: JRXCAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. -----

KIND -----

DATE -----

APPLICATION NO. -----

DATE -----

JP 1983-146694

19830812

AB polyolefins prepared by polymerizing C₃-C₈ olefins in the presence of a solid catalyst component containing Mg, halogen, and Ti, an organoaluminum compound, and an aromatic carboxylic acid ester were deodorized by contacting with C₈ alc. vapor. Thus, 15 mmol suspended in 30 mL toluene was treated over 30 min with a solution of 15 mmol MeSi(OEt)₃ in 10 mL toluene at room temperature, heated at 60° for 1 h, cooled to -10°, treated over 30 min with a solution of 27 mmol BuMgCl in diisopropyl ether, and heated at 30° for 1 h to obtain a carrier. The carrier was suspended in 30 mL toluene was heated with 150 mmol TiCl₄ and 6.5 mmol EtOBz at 90° for 1 h and filtered hot to obtain a solid product which was then suspended in toluene heated with 150 mmol TiCl₄ at 90° for 1 h, and filtered hot to obtain a solid catalyst component (I) with Ti content 2.8%. A heptane suspension of 16 mg I, 0.24 mmol Me₂CC₆HMe-P in 10 mL heptane, and 0.94 mmol Et₃Al in 4 mL heptane were mixed and autoclaved with 1200 mL liquid propane at 65° for 1 h to obtain 250 g powdered polypropylene [9003-07-0] with boiling heptane-insol. content 94.9%. The polymer (200 g) fluidized with N was contacted with 120 g 2-propanol [67-63-0] vapor at 110° for 30 min to obtain 200 g substantial deodorization.

15. ANSWER 23 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1980:01359 CAPLUS

DOCUMENT NUMBER: 92:41359

TITLE: Supported catalyst for producing unsaturated esters from C₃-C₁₀ olefins, carboxylic acids, and oxygen in the gas phase

Wunder, Friedrich A.; Quadflieg, Therese; Roscher, Guenter; Heck, Gunter

Roechtl A.-G., Fed. Rep. Ger. Offen. 19 pp.

SOURCE: CODEN: GXXXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. -----

KIND -----

DATE -----

APPLICATION NO. -----

DATE -----

DE 1978-2811211

A1 19790927

DE 1978-2811211

19780315

19780315

PRIORITY APPLN. INFO.: DE 1978-2811211 A 19780315
AB The efficiency of catalysts for the title reaction was improved by using catalyst supports consisting of particles with star-shaped cross-sections or ribbed particles (vs. pellets or Rasching rings). Thus, 1377 g SiO₂ particles (5-pointed star cross-section), point ht. 1.7 mm, diameter 6 mm, bulk d. 0.27 kg/l were treated with 63 g Pd(OAc)₂, 63 g HOAc, and 50 g Bi(OAc)₃ in 1093 mL HOAc and 13 g Ba aurate and 84 g H₂O in 393 mL HOAc and the catalyst was dried at 60°/270 mbar under N. The catalyst gave 963 g/L h conversion of propene and HOAc to allyl acetate, and 648 g/L h conversion of isobutene and HOAc to methylallyl acetate. Data for runs with 4 conventional supports were also given.

L5. ANSWER 24 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1978-406804 CAPLUS
DOCUMENT NUMBER: 89:604
TITLE: Unsaturated esters
INVENTOR(S): Klasse, Donald L.
PATENT ASSIGNEE(S): Union Oil Co., USA
SOURCE: U.S. 6 pp.
DOCUMENT TYPE: USXXAM
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:
PATENT NO. -----
KIND -----
DATE -----
APPLICATION NO. -----
DATE -----
US 1976-659198
1961-141832
A2 1961-141832
A1 19670925
AB Olefins are oxidized to unsatd. esters of organic acids or acetals and/or acetoxy and/or unsatd. ethers of alcs. by contacting the olefin with an organic acid or alc. reactant in the presence of a catalyst comprising a compound of a Group VIII noble metal and a redox agent; the catalyst is kept active by contacting it with O. Thus, C2H₄ [74-85-1] vapor was passed through liquid HOAc [64-19-7] to vaporize some of the HOAc and the resulting mixture was passed through a catalyst bed consisting of PdCl₂, HCl, and CuCl₂ supported on silica. Vinyl acetate [108-05-4] was removed as product from the effluent gas stream, and the unreacted C₂H₄ was recycled to the reactor. The flow of C₂H₄ and HOAc to the reactor was interrupted periodically, and a stream of O was passed through the catalyst bed to regenerate the catalyst.

L5. ANSWER 25 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1977-161909 CAPLUS
DOCUMENT NUMBER: 86:161909
TITLE: Catalysts for organic acetate production
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.
SOURCE: JAXXAD
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
PATENT NO. -----
KIND -----
DATE -----
APPLICATION NO. -----
DATE -----
JP 1967-527769
CA 1964-903693
BE 1964-648814
NL 1964-6336
A 19641204
B4 19760730
A1 19740910
A 19641207
A 1963-59909
A 19630604
AB Pd salt supported by a catalytic carrier is reduced to

Pd metal in an aqueous alkaline solution containing reducing agent. The carrier is then impregnated with alkali and/or alkaline earth metal acetates and dried to give a catalyst for gas-phase production of organic acetates from a mixture of olefin, O₂ and HOAc. Thus, Al2O3 catalyst with aqueous PdCl₂ carriers (80 mg/g surface area) were impregnated with aqueous NH₄ solution, then immersed in an alkaline NH₄ solution. The carriers contained 2 weight % Pd at this stage. The carriers were then impregnated with aqueous solution containing LiOH (12 weight parts/100 weight parts final catalyst) and dried at 150° under vacuum to give catalysts. A 500 cm³ catalyst sample was then placed in a 1500 mm long tube (22 mm inner diameter) and a mixture of ethylene 2.08, HOAc 1.83, and O₂ 0.42 mol/hr was passed through the tube (at 135° and 1 atmospheric). 18% of the ethylene was converted with 87.3% of the products as vinyl acetate and 12.7% of the products as CO₂.

L5 ANSWER 26 OF 52 CAPIUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 197161905 CAPIUS

DOCUMENT NUMBER: 86:161905

TITLE: Silica catalytic supports

INVENTOR(S): Kawai, Kohichiro; Fujiwara, Yuzuru; Nakamura, Michihiko

PATENT ASSIGNEE(S): Kuraray Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.

CODEN: JXXXAF

Patent

Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. ----

KIND ----

DATE ----

APPLICATION NO. ----

DATE ----

PRIORITY APPN. INFO.: JP 1975-79804

AB Sintered silica having a surface area of 150-600 m²/g, total pore volume of 0.3-3.0 ml/g, volume of 40-60 Å diameter pores 0.1-1.2 ml/g, volume of 800-1500 Å diameter pores 0.1-0.5 ml/g, and bulk d. 0.2-0.55 g/ml is useful as catalyst carrier. The supports are especially useful for catalysts used for gas phase synthesis of unsatd. esters from olefins. Thus, Aerogel (from Fuji Davison Chemical Co.) was sintered 4 h at 900°, pulverized to give SiO₂ powder (4 μ average diameter, porosity 0.65 ml/g, surface area 210 m²/g, average micro pore diameter 112 Å, bulk d. 0.50 g/ml), 5 kg of which was mixed with 9 L of aqueous 10 weight % SnO₂-N (silica gel from Nissan Kagaku Kogyo K.K.), and the mixture was made into 5-mm-diameter spherical pellets, the pellets were dried at 100° (5 h) and sintered at 700° (4 h) to give catalytic support whose surface area, total pore volume, small pore (40-60 Å) volume, large pore (1000-1300 Å) volume, bulk d., and compressive strength were 252 m²/g, 1.44, 0.6, 0.25 mL/g, 0.38 g/mL, and 14.0 kg/pellet, resp. The support was then impregnated with Pd 3.3, Au 1.5, and KOAc 30 g/L-silica, and vinyl acetate was synthesized from a mixture of ethylene 60, O₂ 10, HOAc 30 mol. % at 145°, space velocity 800/h (at 0°, 1 atm), and at 1 atm: the rate of vinyl acetate production was 2.80 ton/m³-catalyst-day.

L5 ANSWER 27 OF 52 CAPIUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 1975:605969 CAPIUS

DOCUMENT NUMBER: 83:203969

TITLE: Carboxylic acid ester

INVENTOR(S): Onoda, Takeru; Wada, Keisuke; Otake, Masayuki

PATENT ASSIGNEE(S): Mitsubishi Chemical Industries Co., Ltd., Japan

SOURCE: Ger. Offen., 22 pp.

CODEN: GRXXBX

Patent

DOCUMENT TYPE: FAMILY ACC. NUM. COUNT: 1

LANGUAGE: German
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO. ----

KIND ----

DATE ----

APPLICATION NO. ----

DATE ----

PRIORITY APPN. INFO.: DE 2510089

AB 50121191

JP 56034345

JP 51008188

JP 56021463

NL 7502572

FR 2263399

GB 1462749

A 19770126

A 19740710

PRIORITY APPN. INFO.: JP 1974-79007

AB Aromatic compds. or olefins were converted into esters by reaction with O₂ and HOAc in the gas phase in the presence of a catalyst consisting of Pd, Sb and K or Zn acetate. The catalyst support (SiO₂) was first soaked in an aqueous solution of Sb compound (e.g., SbCl₃), dried, and calcined in a stream of O₂, then soaked in Pd salt and HOAc salt solution, dried and calcined. Reaction of C6H₆ with O and HOAc in the presence of these catalysts gave PhOC.

L5 ANSWER 28 OF 52 CAPIUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 1975:98814 CAPIUS

DOCUMENT NUMBER: 82:98874

TITLE: Carbon-chain polymers

INVENTOR(S): Ponomareva, A. P.; Ponomareva, T.

I. E.; Markovskii, P. E.; Raspopov, L. N.; Rozenberg, B.

A.; Dyachkovskii, F. S.

PATENT ASSIGNEE(S): Institute of Chemical Physics, Chernogolovka, USSR

SOURCE: U.S.S.R. From: Otrytiyiva, Isobet., Prom. Obrazets, Tovarnyye Znaki 1974, 51(33), 74.

DOCUMENT TYPE: Patent

LANGUAGE: Russian

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. ----

KIND ----

DATE ----

APPLICATION NO. ----

DATE ----

PRIORITY APPN. INFO.: SU 1975-79804

A 19750627

AB Sintered silica having a surface area of 150-600 m²/g, total pore volume of 0.3-3.0 ml/g, volume of 40-60 Å diameter pores 0.1-1.2 ml/g, volume of 800-1500 Å diameter pores 0.1-0.5 ml/g, and bulk d. 0.2-0.55 g/ml is useful as catalyst carrier. The supports are especially useful for catalysts used for gas phase synthesis of unsatd. esters from olefins. Thus, Aerogel (from Fuji Davison Chemical Co.) was sintered 4 h at 900°, pulverized to give SiO₂ powder (4 μ average diameter, porosity 0.65 ml/g, surface area 210 m²/g, average micro pore diameter 112 Å, bulk d. 0.50 g/ml), 5 kg of which was mixed with 9 L of aqueous 10 weight % SnO₂-N (silica gel from Nissan Kagaku Kogyo K.K.), and the mixture was made into 5-mm-diameter spherical pellets, the pellets were dried at 100° (5 h) and sintered at 700° (4 h) to give catalytic support whose surface area, total pore volume, small pore (40-60 Å) volume, large pore (1000-1300 Å) volume, bulk d., and compressive strength were 252 m²/g, 1.44, 0.6, 0.25 mL/g, 0.38 g/mL, and 14.0 kg/pellet, resp. The support was then impregnated with Pd 3.3, Au 1.5, and KOAc 30 g/L-silica, and vinyl acetate was synthesized from a mixture of ethylene 60, O₂ 10, HOAc 30 mol. % at 145°, space velocity 800/h (at 0°, 1 atm), and at 1 atm: the rate of vinyl acetate production was 2.80 ton/m³-catalyst-day.

L5 ANSWER 29 OF 52 CAPIUS COPYRIGHT 2006 ACS ON STN

ACCESSION NUMBER: 1975:90666 CAPIUS

DOCUMENT NUMBER: 82:90466

TITLE: Catalyst for gaseous production unsaturated esters containing titanium (oxide), palladium, alkali metal carbonylate and a carrier

INVENTOR(S): Nakamura, Seishiro; Kushida, Koichi; Yasui, Akio

Kurayay Co., Ltd.

SOURCE: Jpn. Tokkyo Kono, 3 pp. Division of Japan. 72 45,324 (CA '78: 57793a).

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

- PATENT INFORMATION:
- | | | | | |
|------------|-------|----------|-----------------|----------|
| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
| ----- | ----- | ----- | ----- | ----- |
| JP 4903809 | B4 | 19740816 | JP 1972-17673 | 19720221 |
- PRIORITY APLN. INFO.: AB A catalyst for gas-phase ester manufacture contains 0.1-10 Pd metal, 0.1-20 alkali metal carboxylate, and 0.01-50 weight % Ti metal or oxide, with optional Au or Ag additives, on an active C, Al2O3, SiO2, or aluminosilicate carrier. The Ti is added to the known Pd catalyst by conventional methods. The esters, e.g., vinyl acetate, are prepared by reaction of alkene with carboxylic acid and oxygen at 80-200° under 1-10 atmospheric pressure.
- L5 ANSWER 30 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1974:412587 CAPLUS
DOCUMENT NUMBER: 81:12887
TITLE: Organic esters
INVENTOR(S): Nakamura, Seishiro; Yasur, Teruo
SOURCE: Kuraray Co., Ltd.
PATENT ASSIGNEE(S): Jpn. Kokai Tokyo Koho, 6 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
- | | | | | |
|-------------|-------|----------|-----------------|----------|
| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
| ----- | ----- | ----- | ----- | ----- |
| JP 48103509 | A2 | 19731225 | JP 1972-35553 | 19720407 |
- PRIORITY APLN. INFO.: AB Olefins, esters of unsatd. ales., or alkylbenzenes were treated with O and carboxylic acids to give organic esters in the presence of a Pd catalyst on a carrier, in which the volume of pores having a diameter \geq 1000 Å occupied 28% of the total volume of pores. Thus, a gas mixture containing butadiene, O, AcOH, and N (30:10:20:40) was passed at 140° and 5.1/hr over a Pd catalyst on an alumina carrier, in which the volume of pores with a diameter \geq 1000 Å occupied 98% of the total pore volume, to give 1,4-diacetoxy-2-butene (I), 3,4-diacetoxy-1-butene (II), butadienylacetone (III), resp., at 52, 2, and 3 g/catalyst 1 hr after 1 hr reaction. Similarly, with a silica carrier methacryl acetate and 2-methylen-1,3-diacetoxy-propane were formed from a mixture of isobutylene, O, AcOH, and N. Also, 1-butene, 2-butene, AcOH, and air gave I, II, 3-acetoxy-1-butene, 1-acetoxy-2-butene, and III. Similarly, 1-acetoxy-2-butene, AcOH, O, and N gave I, II, and III.
- L5 ANSWER 31 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1974:427889 CAPLUS
DOCUMENT NUMBER: 80:122889
TITLE: Identification of hydroxy(factoxy)alkanes by gas liquid chromatography combined with ir and NMR spectroscopy
AUTHOR(S): Nikitina, N. S.; Mysak, A. E.; Veretenova, T. N.; Tikhonov, V. P.; Lebedev, E. V.
CORPORATE SOURCE: Vses. Nauchno-Issled. Proektno-Konstr. Inst. Neftpererabotka i Neftekhim. Prom., Kiev, USSR
SOURCE: Neftpererabotka i Neftekhim. Prom. (1973), No. 9, 88-91
CODEN: NEEFBY; ISSN: 0548-1406
DOCUMENT TYPE: Journal
LANGUAGE: Russian
AB During the preparation of C8-18 1,2-alkanediols (I) from a-
- olefins by the Prileschaev reaction, the 1-acetoxy-2-hydroxalkane (III) and 1-hydroxy-2-acetoxyalkane (III) intermediates and I were separated by gas chromatog. on a 5% SE-30/Chromosorb W column by using He carrier gas and a flame ionization detector. For all compounds, the retention time increased with increasing C number. On the basis of ir and NMR spectra, the order of elution was I, III, and II of each group with a given C number.
- L5 ANSWER 32 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1974:15437 CAPLUS
DOCUMENT NUMBER: 80:15437
TITLE: Unsaturated esters
INVENTOR(S): Saito, Toshihiko; Takano, Misao; Moriyma, Shigeru; Murayama, Hiroshi
SOURCE: Tekkosha Co., Ltd.
Jpn. Kokai Tokyo Koho, 4 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
- | | | | | |
|-------------|-------|----------|-----------------|----------|
| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
| ----- | ----- | ----- | ----- | ----- |
| JP 48049711 | A2 | 19730713 | JP 1971-84699 | 19711027 |
- PRIORITY APLN. INFO.: AB Vapor phase reaction of a lower unsatd. hydrocarbon with an acid in the presence of O over a palladium [7440-05-3] and gold [7440-57-5] catalyst optionally containing uranium [7440-61-1] and supported on a zinc spinel, gave unsatd. esters. Thus, Al2O3 was saturated with aqueous Zn(NO3)2 solution and heated to form a spinel which was soaked with aqueous HCl containing PdCl2, AuCl3, and U(OAc)3. The treated spinel was treated further with alkaline NaH4·H2O and aqueous AcOK.
- Pd 1, Au 0.1, U 0.1, and AcOK 4.7 weight%. A mixture of propylene [115-07-1] 0.914, O 0.41, and acetic acid [62-19-7] 0.53 mole/hr was passed over the catalyst at 160.deg. to give allyl acetate [591-87-7]. Vinyl acetate [108-05-4], vinyl propionate [105-38-4], and allyl propionate [2408-20-0] were similarly prepared.
- L5 ANSWER 33 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1972:526021 CAPLUS
DOCUMENT NUMBER: 77:126021
TITLE: Silica supports for palladium catalysts for olefin oxacyclation
INVENTOR(S): Fernholz, Hans; Wunder, Friedrich; Schmidt, Hans Joachim Farberke Roehst A.-G.
PATENT ASSIGNEE(S): Ger. Offen., 12 pp.
SOURCE: CODEN: GMXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
- | | | | | |
|------------|-------|----------|-----------------|----------|
| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
| ----- | ----- | ----- | ----- | ----- |
| DE 2100778 | A | 19720720 | DE 1971-2100778 | 19710106 |
- DE 2100778 B2 19771117 NL 1972-13 19720103
NL 7200013 A 19720111 ES 1972-398552 19720103
ES 398552 A 19740816 ZA 19729927 2A 1972-37 19720104
ZA 7200037 A 19730705 AU 1972-37544 19720104

IT 946346 A 19730521 IT 1972-19094 19720105
 CH 555797 A 19741115 CH 1972-111 19720105
 CH 164760 P 19740411 HU 1972-H01447 19720105
 GB 1313272 A 19741106 GB 1972-636 19720105
 SU 19760991 D 19760415 SU 1972-153287 19720105
 AT 312571 B 19740110 AT 1972-115 19720107
 PL 83922 B 19741031 PL 1972-12807 19720107
 SE 39264 A 19751031 SE 1972-103 19720107
 CS 176180 P 19770630 CS 1972-103 19720107
 NO 156925 B 19770822 NO 1972-29 19720107
 JP 57005215 B4 JP 1972-4556 19720107
 BE 5777880 A1 BE 19720129 19720110
 FR 2121633 A5 FR 19720825 19720110
 FR 2121633 B1 19770715 19721006
 RO 56637 P 19750915 RO 1972-63312 19730622
 US 3339199 A 19760217 US 1973-312686 19730622
 PRIORITY APPLN. INFO.: A2 19710106
 SOURCE: DE 1971-2100778 A 19710108

AB Pd catalyst supports for the manufacture of RIC02R (R = CH₂:CH, CH₂:CHCl₂, or CH₂:CMeCH₂; R₁ = Me or Et) from RH and RIC02H in gas phase in the presence of SiO₂ (sp. surface area 205 m²/g, pore volume 0.95 ml/g, and <10⁸ pores of diameter <30 Å). Thus, 850 g. C2H₄/hr, 75 l. O/hr and 870 g. HOAc/hr were passed over a catalyst (from 1 l. of above support, Pd acetate 10.7, Cd(OAc)₂ 1.9, and AcOK 20 g) to give 305 g. MeC02CH₂/hr as compared with 220 g/hr for SiO₂ supports of sp. surface area 160 m²/g, pore volume 0.73 ml/g, and pellet size 6 mm.

L5 ANSWER 34 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
 DOCUMENT NUMBER: 1970-467084 CAPLUS
 LANGUAGE: 73:67084

TITLE: Preparation of unsaturated carboxylic esters by olefin oxidation
 INVENTOR(S): Sennewald, Kurt; Vogt, Wilhelm; Erpenbach, Heinz;
 Glaser, Hermann
 PATENT ASSIGNEE(S): Knapack A.-G.
 SOURCE: Ger. Offen., 15 pp.
 CODEN: GWXXBX

PATENT NO. ---- DATE APPLICATION NO. ---- DATE
 DE 1808610 A 19700611 DE 1968-1808610 19681113

DE 1808610 B2 19740822 19681113
 DE 1808610 C3 19750410 19681113
 CH 517692 A0 19720115 CH 1968-517692 19681030
 BR 6913955 A 19730315 BR 1968-233955 19681106
 NL 6916848 A 19700515 NL 1969-513096 CAPLUS
 GB 1250265 A 19711020 GB 1968-150265 19681107
 CS 164835 P 19751128 CS 1968-7395 19691110
 ES 373419 A1 19711216 ES 1968-373419 19691111
 AT 289742 B 19710510 AT 1968-16069 19691112
 SE 371433 B 19741118 SE 1968-15523 19691112
 NO 132864 B 19751013 NO 1968-488 19691112
 RO 59890 P 19760615 RO 1968-61551 19691112
 BE 741636 A 19700513 BE 1968-711636 19691113
 FR 2024839 A5 19700903 FR 1968-39989 19691113
 JP 5100485 B4 19760625 JP 1968-91062 19691113

PRIORITY APPLN. INFO.: DE 1968-1808610 A 19681113
 AB Viny. acetate (1), propionate, or isobutyrate and alkyl acetate are prepared from **olefins**, C₂-20 aliphatic and aromatic **carboxylic acids** and mol. O with a metal ion catalyst and catalyst activators.

Thus, a carrier of silicic acid globules was ground in a solution of PdCl₂ and HgCl₄, dried, and treated with aqueous hydrazine hydrate. A gaseous mixture of AcOH, N₂O, and C2H₄ was preheated with KOH and NaOAc on silicic acid and passed over the heated catalyst to give a condensate containing 91% I. This method gave 7.1% C2H₄ conversion and high catalyst efficiency, while a method in which the gas mixture was not pretreated with HOAc and NaOAc gave lower catalyst efficiency and 0.23% C2H₄ conversion.

L5 ANSWER 35 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
 DOCUMENT NUMBER: 1970-45664 CAPLUS
 LANGUAGE: 73:55664
 TITLE: Unsaturated esters of carboxylic acids
 INVENTOR(S): Sennewald, Kurt; Vogt, Wilhelm; Glaser, Hermann;
 Erpenbach, Heinz; Ohorodnik, Alexander; Joest, Herbert
 PATENT ASSIGNEE(S): Knapack A.-G.
 SOURCE: Ger. Offen., 10 pp.
 CODEN: GWXXBX

DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:
 PATENT NO. ---- KIND DATE APPLICATION NO. ---- DATE
 DE 1804347 ---- A 19700618 DE 1968-1804347 19681022
 DE 1804347 C3 19730222
 SU 438116 D 19740730 SU 1969-1362726 19690917
 NL 6915303 A 19704424 NL 1968-15303 19681009
 AT 289740 B 19710510 AT 1969-3857 19681020
 ZA 6907398 A 19710331 ZA 1969-7398 19691021
 GB 1235632 A 19701616 GB 1969-1235632 19691021
 BE 740637 A 19704422 BE 1968-740637 19681022
 FR 2030060 A5 19701030 FR 1968-36257 19681022
 PRIORITY APPLN. INFO.: DE 1968-804347 A 19681022
 AB Unsatd. esters of carboxylic acids were prepared by reaction of diolins with carboxylic acids in the presence of O and a Pd catalyst at 180-90°/8 atmospheric. Thus, 1350 g. SiO₂ carrier of 0.1-0.2 mm particle size was mixed with 16.1 g. PdCl₂ and 5.85 g. HgCl₂, dried at 80° under N, added to alkaline N2H4 solution, mixed with 125 g. EtCO₂ in H₂O, and dried at 40-60° in vacuo to give a catalyst containing 18 Pd, 0.4% Au, and 3% K. A gas containing 64% ethylene, 16% EtCO₂, 8% O, and 12% CO₂ was passed over the above fluidized bed catalyst at 190°/8 atm at 34 cm/sec and 4.1 sec contact time to give 92% EtCO₂:CH₂, based on 8.5% ethylene reacted. Similarly prepared were 91% iso-PrCO₂:CH₂, based on 6.5% ethylene reacted, and 85% AcCO₂:CH₂, based on 5.2% CH₂:CHMe reacted, resp.

L5 ANSWER 36 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
 DOCUMENT NUMBER: 1969-513096 CAPLUS
 LANGUAGE: 71:113096
 TITLE: Minor acidic constituents of vetiver oil. II. Cyclcopacamphenic and epicyclopacamphenic acids
 AUTHOR(S): Kido, Fusao; Sakuma, Reiko; Uda, Hisashi; Yoshikoshi, Akira
 CORPORATE SOURCE: Tohoku Univ., Sendai, Japan
 SOURCE: Tetradeuron Letters (1969), (37), 3169-72
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 GI For diagram(s), see printed CA Issue.
 AB The 2 minor acids isolated from vetiver oil were separated as Me esters by preparative gas liquid chromatog. of 68 lowest boiling fraction, b2 112-18°, of the whole ester mixture

and submitted to mild alkaline hydrolysis to yield the major acid, cyclocapromaphenic acid, C15H22O2 (I, R = CO₂H) (II), m. 151.5-25°, [α]_D19D -14.7° (c 1.25, CCl₃), and the minor acid epicyclo-copacromaphenic acid (I, R = CO₂H) (III), m. 161.0-8.5°, [α]_D20-5D 78.3° (c 3.6, CCl₃). Spectral and chemical evidence indicated that I are saturated tetracyclic compds.

with one secondary and 2 tertiary Me groups with a probable tricyclic carbon framework, both derived from the same parent hydrocarbon I (R = Me) (IV). The mixed acids treated with Pb(OAc)₄-LiCl followed by dehydrochlorination of the epimeric Cl derivs (I, R = Cl) gave a mixture of geometrically isomeric **olefins** (V). The N.M.R. chemical shift of the values for the secondary Me group in the spectra of the Cl derivs. and V showed the existence of the >CH₂CH₂CO₂H group in II and III. V hydroxylated and submitted to glycol cleavage gave a single cyclic ketone (VI), ν 1701, 1420 cm.⁻¹. The Baeyer-Villiger oxidation of VI afforded an **ε-lactone**, C12H16O₂, m. 109-11°, subsequently transformed into an oxo **carboxylic acid** (VII), C12H16O₃, m. 114.0-15.5°, by saponification followed by Jones oxidation. The spectral properties of VII and its Me ester verified the tricyclic nature of the 3-carbon side chain would be that the C skeleton of I and II, including the 3-carbon side chain would be that the C skeleton of IV-VII, R = iso-Pr, R1 = H) (IX). According to the reaction sequence: -CO₂Me → -CH₂OH → -Me, II and III gave the same hydrocarbon IV whose ir and N.M.R. spectra indicated stereoisomeric with IX with respect to the iso-Pr group, a supposition confirmed by conversion of II and III to IX by deacetoxybifunctional acetoxylation to I (R = OAc) with Pb(OAc)₄; hydrolysis with alc. KOH followed by Jones oxidation to VII (R = H, R1 = Ac); epimerization with NaOEt to yield the isomer VIII (R = Ac, R1 = H); and Wittig reaction with Ph₃P·CH₂CO₂H to VIII (R = MeC₂CH, R1 = H), followed by catalytic hydrogenation over (Ph₃P)·3RhCl as catalyst. The resulting hydrocarbon C15H₂₄, [α]_D20D 67.8° (c 1.15, CCl₃), M_r204 was spectroscopically identical with (-)-cyclosativene (IX), [α]_D 94.1°, leading to the structures of II and III. The strongly pos. sign of the Cotton effect in the O.R.D. of VI supported the absolute stereochemistry of the C skeleton of VI.

15 ANSWER 37 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN

1969:402987 CAPIUS

DOCUMENT NUMBER: 71:2987

TITLE: Esters of unsaturated alcohols

INVENTOR(S): Capp, Clifford W.; Durston, Peter J.

SOURCE: BP Chemicals (U.K.) Ltd.

BRIT., 2 PP.

CODEN: BRXXXA

PATENT: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO.

----- ----- -----

GB 1148583 19690416 1966-28391

AB Vinyl acetate (I) is manufactured by combining C2H₄, O, and AcOH in the vapor phase at elevated temps. in the presence of Pd deposited on Al₂O₃, Fe oxide, or Al Mg spinel **supports**. The exhausted

catalysts are reactivated by treatment in 2N NaOH. Thus, 9-16-mesh Al₂O₃ was treated with 1:1 PdCl₂-HCl solution, dried at 140°, and reduced with alkaline NaH₄ to yield 2% Pd/al₂O₃ catalyst (II). C2H₄ 0.29, O 0.06, and AcOH 0.14 mole/l/hr. were combined at 140° in the presence of II to yield 0.21 mole/l/hr. I after 4 hrs. and 0.13 mole/l/hr. For the next 190 hrs., before falling to 0.02 mole/l/hr. arter 217 hrs. The spent II was treated with 3 successive ams. of 2N NaOH, followed by 6 successive washings with cold H₂O, and drying at 140° to give a reactivated-II that yielded 0.18 mole/l/hr. I for the next 256 hrs. Uses of other

Pt-group metals on aluminosilicates, heavy metal oxides, metal oxide-Al₂O₃ complexes, carbon, or pumice, and catalyst reactivation with aqueous solns. of KOH, Na₂CO₃, or K₂CO₃ and aq. solns. of Na-(or K) methoxide or methoxide, were claimed.

15 ANSWER 38 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN

DOCUMENT NUMBER: 70:116669 CAPIUS

TITLE: Finely divided metals of the platinum group

PATENT ASSIGNEE(S): Monsanto Co.

SOURCE: Brit., 7 pp.

CODEN: BRXXXA

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO.

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GB 1146530 19690326 GB 1966-22105

DE 1533102 CH DE

PRIORITY APPN. INFO.: 19650518

AB A method of producing finely divided metals of the Pt group, the products obtained, **supported** catalysts made therefrom and their use for hydride generation and dehydrogenation are described. The finely divided metal is obtained by reducing a solution of a salt of the metal in the presence of an olefin, acetal, ketone, ether, carboxylic acid **ester**, or olefin, which has a reducing action on the salt and forms a soluble complex with the metal, by the action of H₂ on the solution in the absence of a borohydride, or by thermally decomposing the complex, thereby producing the metal in a zero-valent state. The process may be carried out in the presence of a nonreducing solvent for the metal salt.

Particularly suitable reducing agents (solvents) for this process are the cyclic ethers, especially propylene oxide. In carrying out the treatment with H₂, the solution may be preheated. The thermal decomposition of the complex may

be effected before or after removal of excess reducing agent and may occur slowly at room temperature; usually it is sufficient to heat the solution for 10 to 120 min. up to 90° to obtain a quant. suspension of the Pt. metal.

The finely divided Pt metals can be deposited on supports, especially Al₂O₃ which have been subjected to ph. or chemical pretreatment such as glowing, action of acids, or with superheated H₂O-vapor. The metals are established on the support by steeping or by pouring the solution onto the support. Solvent is then removed in vacuo at the room temperature with effective stirring. The metal compound which remains on

the support contains enough, probably complex-linked, reducing agent to give the zero-valent metal on heating up to higher temps. (e.g., to 150-250° for hydrogenation catalysts) preferably under reduced pressure. For example, to prepare finely divided Pt on a support, the pulverized or granulated support is heated in a vacuum (0.004 mm.) at 150° for 2 hrs. The Pt salt is subject to the same treatment. A solution containing (1-2) + 10-2 mole Pt salt/mole solution is prepared in an atmospheric of Ar by using the cited reducing agent. The solution is slowly added, with cooling, either to the cooled powdery support or to the granulated support previously freed of air by evacuating until the carrier mass is well saturated with it. The excess solvent is distilled immediately with stirring in a vacuum at 25° and the catalyst is heated in the vacuum at the indicated aftertreatment temperature for 1 hr. After cooling the catalyst to room temperature,

air is admitted. The catalyst is used approx 24-48 hrs. later. The activities of such catalysts prepared with different reducing agents and

supports are tabulated.

L5	ANSWER 39 OF 52	CAPIUS	COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER:	1969:105966	CAPIUS	
DOCUMENT NUMBER:	70:103986		
TITLE:	Carboxylic acids and their unsaturated esters		
INVENTOR(S) :	Sennwald, Kurt; Vogt, Wilhelm; Erpenbach, Heinz;		
PATENT ASSIGNEE(S) :	Glaeser, Hermann		
SOURCE:	Knappeck A.-G. S. African, 18 pp. CODEN: SEXXAB		
DOCUMENT TYPE:	Patent		
LANGUAGE:	English		
FAMILY ACC. NUM. COUNT:	1		
PATENT INFORMATION:			
PATENT NO.	KIND	DATE	APPLICATION NO.
ZA 6802010	-----	19680829	DE 19680322
DE 1618591	DE	FR	19680322
FR 1567312	FR	GB	19680322
GB 1199091	GB	RO	19680322
RO 52442	RO	US	19680322
US 3631079	US	US	19680322
PRIORITY APPN. INFO.:		DE	19670405
AB	An aldehyde is partially or completely substituted for its corresponding acid in the gas -phase reaction of an olefin , the carboxylic acid, and O in contact with a Pd-containing carrier catalyst at elevated temperature to give an unsat. ester of the acid. The aldehyde is simultaneously oxidized in the process, thus reducing the cost of oxidation of the aldehyde to the acid in an addnl. step. Thus, the catalyst was prepared by impregnating 1 kg. of a ball-shaped silicic carrier with aqueous solution of 8 g. PdCl ₂ and 3 g. H ₂ O ₂ , drying the mixture with agitation, pouring into a 4-5% H ₂ NNH ₂ .H ₂ O solution at 40°, and, after reduction was complete, pouring off the supernatant liquid, washing the catalyst with distilled H ₂ O, impregnating with 15% aqueous KOAc solution, decanting the solution, and drying the catalyst under reduced pressure at 60°. The catalyst (1 l.) was placed in a steel tube, and 500 1./hr. C ₂ H ₄ , 50 g./hr. HOAc, and 25 g./hr. Ach was passed over the catalyst at 6 atmospheric absolute. The reaction gas was cooled to 0° to give a mixture comprising 70% (428 g.) HOAc, 23.4% vinyl acetate, 6.45% H ₂ O, and approx. 0.2% Ach. Approx. 32.1 g. fresh HOAc was formed by Ach oxidation, and approx. 0.9% of the HOAc obtained by oxidation reacted. Vinyl isobutyrate was similarly prepared		
L5	ANSWER 40 OF 52	CAPIUS	COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER:	1969:96179	CAPIUS	
DOCUMENT NUMBER:	70:96179		
TITLE:	Vinyl acetate from saturated organic acids and olefins		
PATENT ASSIGNEE(S) :	Stamicarbon N. V. Addn. to Neth. Appl. 66 08559		
SOURCE:	Neth. Appl., 7 pp.		
DOCUMENT TYPE:	Patent		
LANGUAGE:	Dutch		
FAMILY ACC. NUM. COUNT:	1		
PATENT INFORMATION:			
PATENT NO.	KIND	DATE	APPLICATION NO.
NL 6706640	-----	19681113	NL 19670512
DE 1643077	DE	DE	

GB	GB 1154517	Catalysts useful for preparing unsat. esters from saturated organic acids, olefins , and mol. O are prepared by impregnating a carrier with a solution of a Pd salt and Pt, or Rh salt, and then drying, hydrolyzing, and reducing the impregnated carrier mass. Thus, catalysts are prepared by impregnating Al2O3 with salts and Pt salts. The impregnated mass is then dried, hydrolyzed by treatment with boiling NaHCO ₃ solution, and reduced. These catalysts are used in the synthesis of vinyl acetate (I) by passing 120 l./hr./l. of catalyst of a 1:1:0.4 molar C ₂ H ₄ -HOAc- gas mixture over the catalyst at 1 atmospheric pressure. For example a catalyst containing 2.0% Pd and 0.02% Pt gave a C ₂ H ₄ conversion of 31.6 mole % and an 89% yield in 1 day and 29.8 and 91%, resp., in 18 days. A catalyst containing 2.0% Pd and no Pt gave 15.1% conversion and 92% yield in 1 day and 7 and 90%, resp., in 4 days at 118°. A catalyst containing no Pd and 3.0% Pt gave resp. values of 10.5 and 9% in 1 day at 111°. Best results (42.5 and 88%, resp.) were obtained with 2.2% Pd and 0.05% Pt in 1 day at 120°. A catalyst containing Rh was also used. These catalysts have excellent retention of activity over long operating times, and give good conversions to the desired product.	
L5	ANSWER 41 OF 52	CAPIUS	COPYRIGHT 2006 ACS on STN
	ACCESSION NUMBER:	1969:96171	CAPIUS
	DOCUMENT NUMBER:	70:96171	
	TITLE:	Gas-phase synthesis of vinyl esters from ethylene, oxygen, and carboxylic acids with a palladium-alkali metal acetate catalyst	
PATENT ASSIGNEE(S) :	Kurashiki Rayon Co., Ltd.		
SOURCE:	Brit., 10 pp.		
DOCUMENT TYPE:	CODEN: BRXXXA		
LANGUAGE:	Patent		
FAMILY ACC. NUM. COUNT:	1		
PATENT INFORMATION:	English		
PATENT NO.	KIND	DATE	APPLICATION NO.
-----	-----	-----	-----
GB 1142250	-----	19690205	-----
DE 1568339	DE	DE 1966-56274	-----
FR 1520433	FR	DE	19661215
US 3367767	US	FR	19661209
PRIORITY APPN. INFO.:	JP	JP	19661217
AB	Vinyl esters were prepared by continuous gas phase synthesis from C ₂ H ₄ , O, and an organic carboxylic acid , using a catalyst system having an improved and high catalytic activity and which minimizes the rate of loss of its catalytic activity by performing the reaction under a low partial pressure of O and organic carboxylic acid . Thus, bead alumina having 99.3% Al ₂ O ₃ , a 210 m ² /g. surface area, and 10-14 mesh were calcined 3-5 hrs. in air at 550-1300°. The bead alumina was prepared by adding 175 g. CaCO ₃ wetted with 30°C. water to a solution of 342 g. Al ₂ (SO ₄) ₃ dissolved in 800 cc. water. CO ₂ was generated and CaSO ₄ was precipitated. CaSO ₄ was filtered to give Al ₂ O ₃ (I). The liquid had 3.38 pH. I was dropped through an 85° heated glass tube to give a gel, which was purified and calcined 5 hrs. at 500°. The calcined carrier (35 parts) was added to 50 parts water containing 0.3 part HCl and 1.3 part PdCl ₂ and the system dried. PdCl ₂ was reduced by dropping a reducing liquid on the dried catalyst system. The liquid was prepared by adding 2.5 parts of 80% hydrazine hydrate solution to 50 parts N NaOH solution. The reduced catalyst was water-washed and placed in 10 parts water containing 0.7 parts AcOH and dried. The catalyst (35 g.) was placed in a glass tube heated to 110° and a gaseous mixture containing C ₂ H ₄ , O, AcOH (1:1.2 molar ratio) was passed over the catalyst at 8 l./hr. In an experiment, where the calcination temperature for the carrier was 900° and the surface area of the carrier was 120 m ² /g., the production rate of vinyl acetate (II) was 40.1 g. II/l. /catalyst hr., the II selectivity, 94%, and AcH selectivity, <0.1.		

L5 ANSWER 42 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
 ACCESSION NUMBER: 1996:27122 CAPLUS
 DOCUMENT NUMBER: 64:27122
 ORIGINAL REFERENCE NO.: 6:499d-e
 TITLE: Ethylenically unsaturated esters
 PATENT ASSIGNEE(S): Pullman Inc.
 SOURCE: 5 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PARENT INFORMATION:
 PATENT NO. KIND DATE APPLICATION NO. DATE

 GB 1007815 P 1965/022 US 19601223
 PRIORITY APPLN. INFO.:
 AB Olefins halogenated on one of the doubly bound C atoms react in liquid or vapor phase, under anhydrous conditions, with carboxylic acids in the presence of halides of Group VIII metals to form unsat'd esters. The halides may be used in combination with a support carrier, or diluent, such as alumina, silica gel, and alundum. The greater the ratio of the catalyst to olefin reactant the faster the rate of reaction. Reaction takes place between 70 and 300°, and mostly at the atmospheric pressure. Thus, 3.56 g. Pdc12 and 11.36 g. Na2HPO4 is kept for 2-4 days in vacuo at 37° F., cooled to room temperature, 50 cc. of isooctane added, and the vessel flushed with vinyl chloride at 720 mm. with shaking. The reaction vessel is charged with 3.76 g. AcOH and shaken at room temperature for 4 hrs. The yield of vinyl acetate is 2.6 mole-% on the basis of Pdc12.

L5 ANSWER 43 OF 52 CAPLUS COPYRIGHT 2006 ACS ON STN
 ACCESSION NUMBER: 1996:26837 CAPLUS
 DOCUMENT NUMBER: 64:26837
 ORIGINAL REFERENCE NO.: 64:48819-h, 4888a-h, 4888a
 TITLE: Solvolytic rearrangement of the 2-(1-cyclopentenyl)ethyl system
 AUTHOR(S): Cllosion, W. D.; Kwiatkowski, G. T.
 CORPORATE SOURCE: Columbia Univ.
 SOURCE: Tetradecon (1965), 21(10), 2779-89
 DOCUMENT TYPE: COREN: TETRAB; ISSN: 0040-4020
 Journal
 LANGUAGE:
 GI For diagram(s), see printed CA issue.
 AB cf. CA 62, 6357d. The acetolysis of 2-(1-cyclopentenyl)ethyl bromobenzene-sulfonate (II) was investigated. Reduction of 1-cyclopentenylacetic acid in tetrahydrofuran with LiAlH4 yielded 90% 2-(1-cyclopentenyl)ethanol (III). b3 65-6°, n2D 1.4781. Similar reduction of cyclopentylacetate acid gave 90% yield of 2-cyclopentylethanol (III), b3 77-8°. Conversion of 17 g. II gave 10 g. of the corresponding bromide, b0 5-43-4°. The bromide (57 millimoles) and 70 millimoles KCN in 25 ml. HOCH2CHOH heated 2.5 hrs. at 100° and the cooled solution poured into saturated aqueous NaCl, extracted with Et2O, and the residue on evaporation heated 20 hrs. at 95° in 200 ml. 20% aqueous KOH, the cooled solution washed with aqueous HCl, extracted with Et2O, and the isolated 3-(1-cyclopentenyl) propionic acid, (2.5 g., m. 58-61°) reduced with LiAlH4 in tetrahydrofuran yielded 84% 3-(1-cyclopentenyl)-propanol (IV), b3 66-7°. Preparation according to Alibisetti, et al. (CA 51, 1041d) gave 3-methylcyclohexanol (V), b43 97-5-8°. Zn-Cu couple (12 g.) in 30 ml. dry Et2O and 11.6 millimoles 2-methylenecyclopentanol in 7 g. CH2Cl2 stirred 16 hrs. and diluted with 5 ml. saturated aqueous NH4Cl, the organic layer and Et2O washings washed, dried, and concentrated and the residual oil kept 16 hrs. in 10 ml. saturated MeONa

rearranged acetates seemed unlikely. The first-formed intermediate should account for the rate enhancement and the bicycliconium ion (XVII) would do so and would also explain formation of spiro[2.4]-heptan-4-yl acetate (XVIII) and bicyclo[3.2.0]heptan-1-yl acetate (XIX). The unrearanged 2-(1-cyclopentenyl)ethyl acetate (XX) probably came from a competing solvolytic displacement. The other two rearranged acetates, bicyclo[3.1.0]-hexane-1-methyl acetate (XXXI) and XIV cannot be obtained from XVII and a bridged homoallylic ion (XXII) or a bridge cation (XXIII) were postulated as precursors with conversion to ion (XXXIV) involving only minor changes in bond lengths and conformation. Accordingly the acetate of I was rationalized schematically as: I → XVII → XVIII → XVII (or XXII) → XXIV (or XXIII) → XVI, XVII → XVIII + XVI, XXII (or XXIII) → XVI, XXII (or XXIII) → XIV. Other cations could also be present but their proposed scheme is sufficient to account for the products and to provide a reasonable explanation for the reactivities of the related systems. This general reaction class may have synthetic utility. The biol. extensive 2-[β -indolyl]ethyl system is analogous in many ways to the 2-(1-cyclopentenyl)ethyl structure.

- 15 ANSWER 44 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1966:11686 CAPLUS
 DOCUMENT NUMBER: 64:11686
 ORIGINAL REFERENCE NO.: 64:2169-e-h,2150c
 TITLE: Extension of sugar chains through acetylenic intermediates
 AUTHOR(S): Horton, D.; Hughes, J. B.; Tronchet, J. M. J.
 CORPORATE SOURCE: Ohio State Univ., Columbus
 SOURCE: Chemical Communications (London) (1965), (20), 481-3
 CODEN: CCOMA8; ISSN: 0009-241X
 DOCUMENT TYPE: Journal
 LANGUAGE:
 OTHER SOURCE(S): CASREACT 64:11686
 GI For diagram(s), see printed CA issue.
 AB Oxidation of 1,2:3,4-di-O-isopropylidene- α -D-galactopyranose with MeSO-N,N'-dicyclohexy carbodiimide, followed by ethynylation of the resultant aldehyde gave the 6-epimeric mixture (I). The preponderant epimer (m. 130°) was reduced to the corresponding olefin, m. 106°-08°. Similarly, ethynylation of peri-oxide oxidized 1,2-O-isopropylidene- α -D-glucofuranose gave a mixture of 5-epimers, the D-glucos-epimer gave a crystalline 3,5-dibenzozate, m. 191-3°. Reduction of the latter to the olefin, m. 143-5°, followed by ozonolysis, reduction, and hydrolysis gave glucose. The 3-epimeric heptynes (II) formed by ethynylation of 2,3:5,6-di-O-isopropylidene-aldehyde-L-arabinose were separated by gas-liquid chromatography (g.l.c.) to give the D-gluc- isomer, m. 67-9°, [α]D²² 6.5° (CHCl₃) and the D-manno-isomer, [α]D²²-24 (CHCl₃), in 3:2 proportion: the structures were established by degradative reactions. The 3-acetate of II was treated with bis(1,2-dimethylpropyl)borane (IV) in diglyme, followed by H₂O₂, to give the 2,3-trans-unsaturated aldehyde (III), b.p. 03°. Similarly, ethynylation of 1,2-O-isopropylidene-D-glyceraldehyde gave a 3:2 mixture of 3-epimeric pentone derivs., separable as the 3-acetates (V) by g.l.c. One epimer had [α]D²⁰ -41° (CHCl₃), and the other [α]D²⁰ 50° (CHCl₃). Treatment of V with IV, followed by H₂O₂, gave trans-2,3-dideoxy-4-O-isopropylidene-aldehydo-D-Glycero-2-pentone, the 5-carbon analog of III. Structures given were supported by IR and N.M.R. data.
- 15 ANSWER 45 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1964:084851 CAPLUS
 DOCUMENT NUMBER: 61:84951
 ORIGINAL REFERENCE NO.: 61:1439c-g
 TITLE: Modification of textile fiber properties by radiation-induced graft copolymerization Armstrong, Arthur A., Jr.; Walsh, William K.; Rutherford, Henry A.
- SOURCE: U.S. At. Energy Comm. (1963), NCSC-2477-11, 132 pp.
 DOCUMENT TYPE: Journal
 LANGUAGE:
 AB The modification of fiber properties by exposure to radiation and by graft copolymerization (GP) is described. The radiation source was a 60Co γ -cell 220. The predominant effect of radiation on fibers was degradation. No improvement of the phys. properties of textile fibers by radiation alone was indicated. Vapor-phase GP to textile fibers initiated by γ radiation was investigated for (a) variables affecting the rate of GP; (b) modification of fiber properties by GP; and (c) kinetics and diffusion in the vapor-phase GP process. The higher effects of process variables on the rate of grafting were: (1) higher carrier-gas flow rates increased the rate of grafting; (2) the presence of a 3rd component (e.g. H₂O, MeOH, or AcOH) increased the rate of grafting to acetate and nylon, and was necessary for grafting to cotton, rayon, and wool; (4) O inhibited polymerization; (5) the rate of grafting depended on the fiber and the monomer; the order of reactivity for fibers was acetate > polypropylene > nylon > cotton > wool > polyester > acrylic; the order of reactivity of the monomers studied was Me acrylate > Et acrylate > acrylonitrile > butadiene > vinyl acetate > Me methacrylate > vinyl chloride > styrene; (6) chemical modification of the cellulose increased the rate of grafting to cotton; (7) the lifetimes of free radicals produced by irradiation were long enough to use post-irradiation techniques. Radiation-induced vapor-phase GP of vinyl monomers to textile fibers was studied as a means to improve fiber properties with the following results: (1) cotton was completely resistant to microorganism attack and had increased resistance to heat after grafting with acrylonitrile; (2) polypropylene fiber was dyeable with disperse dyes after grafting with vinyl acetate, Me acrylate, and Et acrylate; (3) nylon and polypropylene had improved weather resistance after grafting with styrene; (4) cotton cloth had improved crease recovery after grafting with allyl acrylate, followed by cross-linking by radiation and grafting with methylacrylamide; (5) nylon had increased dimensional stability at 230° after grafting with allyl acrylate, acrylic acid, or a combination of acrylic acid with a cross-linking monomer, and had 30-50% increased modulus after grafting with acrylic acid alone or in combination with a cross-linking monomer. A preliminary study of the effects of diffusion and kinetics on the vapor-phase GP of acrylonitrile to various textile fibers showed that the effects are complex and cannot be represented by simple math. solutions. Empirical relations were obtained which adequately represented the more complex cases.
- 15 ANSWER 46 OF 52 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1964:124194 CAPLUS
 DOCUMENT NUMBER: 60:22794
 ORIGINAL REFERENCE NO.: 60:4439b-d
 TITLE: Reaction gas chromatography
 AUTHOR(S): Drawert, Friedrich
 SOURCE: Abhandl. Deut. Akad. Wiss. Berlin, Kl. Chem., Geol., Biol. (1962), (1), 9-18
 DOCUMENT TYPE: Journal
 LANGUAGE:
 AB A reactor is interposed between the carrier gas supply and the anal. column in which the sample is transformed chemically to yield better separation of alcs. and polyols in aqueous solution. If the reaction permits, the reactor tube can be placed in the oven compartment in series with the anal. column. A sep. thermostat is used for high-temperature reactions. H₂O can be removed by a C₄H₂ column ahead of the anal. column. Aqueous soins. of lower alcs. are acidified with either tartaric or oxalic acid and passed through a column containing a mixture of NaNO₂ and support material, or a NaVO₂-containing solution of the alcs. is passed through a reactor tube containing one of the acids. The nitrous acid

esters are separated much more readily than the alcs. When alcs., their mixture, or aqueous solns. are sprayed on hot acidic surfaces, yields the corresponding **olefins**, which can be readily separated by distillation. Some alcs. can be determined by first hydrogenating over Raney Ni at 170–200° to the hydrocarbon. Bis(alkyl xanthogenatonickel(II)) complexes are split with dimethylglyoxime, and the alcs. formed by reduction of the **olefin** method. 2-Alkoxyl-2-mercaptopthiazolidines are readily converted to 2-mercaptopthiazolines by splitting off an alc. which is determined as above. Aqueous glycerol is converted by H₂ to iso-PtI, which is determined by propane by the hydrogenation method.

L5 ANSWER 47 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN

DOCUMENT NUMBER: 1963:19840 CAPIUS

ORIGINAL REFERENCE NO.: 58:29840

TITLE: 58:5018d-e

Gas-liquid chromatography of synthetic carboxylic acids and the corresponding alcohols

Vasilescu, V. Inst. Verfahrenstechnik Org. Chem., Leipzig, Germany Abhandl. Deut. Akad. Wiss. Berlin, Kl. Chem., Geol., Biol (1959), (No. 9), 136–53

Journal Available

AB The products of paraffin oxidns., namely C₄–12 carboxylic acids and Cs₁₄ alcs. were separated by using high vacuum grease Number 20 (Carl Zeiss) on ground Kaolin "MKKA" as a carrier. The carboxylic acids were converted to the Me esters with CH₂N₂ and the alcs. were preferably acetylated before analysis.

L5 ANSWER 48 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN

DOCUMENT NUMBER: 1961:101313 CAPIUS

ORIGINAL REFERENCE NO.: 55:19070d-f

TITLE: Catalysts Dowden, Dennis Albert; Caldwell, Alexander M. U. Patent

Patentee: Imperial Chemical Industries Ltd.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. ---- KIND DATE APPLICATION NO. DATE -----

US 971324 ---- 19610328 US -----

AB Catalysts suitable for the oxidation of aromatic hydrocarbons, and other compds., such as paraffins and olefins, each with at least 3 C atoms, aldehydes which yield carboxylic acids, and ketones, to O-containing compds. are processed from molten catalytic vanadyl vanadates (single solid phase or a mixture of solid phases, each phase containing at least 1 of the metals Tl, Ag, K, Na, Li, or Rb, together with the oxides of V where part of the V atoms have a valency of 2 to 4) by impregnation onto a porous, heat-resistant, solid support, such as Al₂O₃, SiO₂, SiC, or firebrick. The preferred catalysts are supported Tl vanadyl vanadate and K; in these the M₂O:V₂O₅ molar ratio, where M is K or Tl, may be 0.01–0.3:1, but is conveniently 0.05:1. Thus, 1.29 g. Ti₂CO₃ and 10 g. of V₂O₅ were fused together and impregnated on particles of α-Al₂O₃ by dipping. A mixture of 21.2 g. O₂-xylene vapor and 480 l. air was passed over 50 ml. of the Tl vanadyl vanadate supported on α-Al₂O₃. At the operating temperature of 500°

the O₂-xylene was converted to phthalic anhydride with a conversion of 64.1% and a pass yield of 35.5%.

L5 ANSWER 49 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1960:1118497 CAPIUS
DOCUMENT NUMBER: 54:1118497
ORIGINAL REFERENCE NO.: 54:22706-a-f
TITLE: Addition of dinitrogen tetroxide to camphene and trans-stilbene: the effect of oxygen
AUTHOR(S): Stevens, Travis E.; Rohm and Haas Co., Huntsville, AL
CORPORATE SOURCE: Journal of the American Chemical Society (1959), 81, 3593–7
CODE: JACSAT; ISSN: 0002-7863
JOURNAL
Unavailable
CASREACT 54:1118497

When an excess of N₂O₄ was added in a dry N₂ stream to an Et₂O solution of camphene, the products isolated were: 5% dl-α-nitrocumphenane (I), m. 64–5°; 35% 3-(nitromethyl)-3-hydroxycumphenane (II), m. 37–9°; 30% 3-(nitromethyl)-3-nitrocumphenane (III), m. 141–3°; and a trace of a nitrate ester, probably 2-nitro-10-nitrocumphane (IV). II (formed by the hydrolysis of 2-nitro-10-nitrocumphenane) and III were the usual products of homolytic reaction of N₂O₄ with **olefins**. Treatment of I and II with solution of KOH in 50% EtOH 30 min. gave I, 59 and 56%, resp. Similarly, base treatment of the crude camphene-N₂O₄ mixture gave I in 40% yield. With O as the **carrier gas**, the camphene-N₂O₄ reaction gave a mixture of products including 3–6% IV, 20–41% 3-isopropylidenecyclopentenyl nitromethyl ketone (V), m. 76–7°, 30% nitroacids of undet. structure, and a total of 6% II or III or both. The structure of V was established by its base-catalyzed cleavage to 3-isopropylidenecyclopentenylcarboxylic acid (VI), m. 50°. VI was found to be identical with camphoic acid (prepared by the hydrolysis of the nitrile produced in the camphene-oxime-AcCl reaction); ozonolysis of VI produced cyclopentanone-3-carboxylic acid, confirming the identity. The UV spectra of V had, in EtOH, 230 m_λ (ish, e 3200), and 316 m_λ (e 660); in 50% EtOH–0.1N NaOH, 234 m_λ (e 4500) and 331 m_λ (e 19,600). When an excess of N₂O₄ was swept with N₂ into an Et₂O solution of stilbene, the products (after hydrolysis of the nitronitrile) included 20.8% meso-α,α'-dimethoxybenzyl, m. 235–7°, 32.2% dl-α,α'-dimethoxybenzyl, m. 149–5°, erythro-α,α'-dimethoxybenzyl, m. 149–5°, erythro-α,α'-dimethoxybenzyl (VII), m. 99–100°, and 12.6% threo-α-hydroxy-α'-nitrobenzyl (VIII), m. 105–5–6.5°. With O as **carrier gas**, the products varied with minor changes in reaction conditions producing from 13.8 to 28.8% mixture consisting of 44% VII and 56% VIII, from 5.4 to 24.9% of a mixture consisting of 65% erythro-α-nitro-α-nitrito-α'-nitrobenzyl (threeo- m. 96°, erythro- m. 165°), from 14.8 to 21.2% α-nitro-α-phenylacetophenone (IX), m. 75.0–5.5°, PhCO₂H, and 0–1.1% PhCHO. Treatment of IX with alc. NaOH produced PhCO₂H and PhCH₂NO₂. It was suggested that the nitrobenzyl radicals reacted with O to form nitroperoxy or nitropoxynitrate radicals.

L5 ANSWER 50 OF 52 CAPIUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1959:121688 CAPIUS
DOCUMENT NUMBER: 53:101688
ORIGINAL REFERENCE NO.: 53:22750g-1,21751a-f
TITLE: Mechanism of the Michaelis-Arbuzov reaction:
olefin formation
AUTHOR(S): Garner, Albert Y.; Chapin, Earl C.; Scanlon, Patricia M.
CORPORATE SOURCE: Monsanto Chem. Co., Springfield, MA
SOURCE: Journal of Organic Chemistry (1959), 24, 532–6

CODEN: JOCEAH; ISSN: 0022-3263

Unavailable

CASREACT 53:121688

AB Report is given for the mechanism of the Michaelis-Arbuzov intermediate. The production of *olacrin* and dialkyl phosphonate, (RO_2PH)₂, is shown to be a general phenomenon when an α -haloalkane which has an activating group on the β -C is treated with a trialkyl phosphite. The formation of these products is explained in terms of an intramol. β -elimination involving the quasi-phosphonium salt intermediate. $\text{PhCH}_2\text{CH}_2\text{Br}$ (92.5 g.), NaI , and 50 mL Me_2CO refluxed overnight gave 92 g. $\text{PhCH}_2\text{CH}_2\text{I}$ (1), b.p. 71°, n_D²⁵ 1.5945.

Triphenoxyn- β -phenyliophosphonium iodide (5 g.) left overnight with excess 10% aqueous NaOH gave 2.5 g. diphenyl phenetyliphosphonate (II), needles, m. 75-6° (hexane). I (73 g.) and 146.2 g. $\text{P}(\text{OEt})_3$ heated 5 days at 128°. The mixture mixed with Et₂O and Me_2CO until it was cream-colored, and the solvent removed gave 28.3 g. triphenoxyn- β -phenyliophosphonium iodide (III), m. 154-7° (sealed tube), gave an immediate precipitate with AgNO_3 which was insol. in dilute HNO_3 .

MeCH_2Br (7.52 g.) refluxed 1.5 hrs. at 131° with addition of 183 g. $\text{P}(\text{OEt})_3$, and heated 3.5 hrs. at 145°, 110.8 g. Et_{Br} removed, and the residue fractionated gave 509.6 g. unreacted dibromide. The first-boiling fraction of 33.7 g. consisted of a mixture of di-Et vinylphosphonate, di-Et phosphonate, and di-Et ethylphosphonate as shown by infrared analysis and vapor phase chromatography. Subsequently 127 g. di-Et β -bromoethylphosphonate (IV), b.p. 90°, n_D²⁵ 1.4564, was obtained. An intermediate fraction (7.7 g.), n_D²⁵ 1.4473, which contained some tetraethyl ethylenediphosphonate, was followed by the sudden evolution of 13.4 g. of material which dropped the head temperature to 30°/2 mm., and had n_D²⁵ 1.4300, shown to be pure di-Et vinylphosphonate (V). IV (50.7 g.) heated to 157° and 35.3 g. distilled $\text{P}(\text{OEt})_3$ (VI) added dropwise in 3 hrs., the heating continued to a total of 6 hrs., and 17.9 g. Et_{Br} removed, and vacuum distilled gave 28 g. of low-boiling material of approx. 64.5% diethyl vinyl phosphonate, 25.8% Et_2 diethyl ethylphosphonate (VII), and 2.9% of unknown material. A higher-boiling fraction consisted of 16.2 g. tetraethyl ethylenediphosphonate (VIII), b.p. 151-7°, n_D²⁵ 1.4397. The acrylic monomer and polymer were identified by their infrared spectra. $\text{PhCH}_2\text{CH}_2\text{Br}$ (90 g.) added dropwise during 3 hrs. to 100 g. Et β -bromopropionate at 155°, the mixture heated 5 hrs., and 48.4 g. Et_{Br} removed during the heating. Vacuum distillation gave 21.9 g. material, b.p. 66-86°, whose infrared spectrum showed it to be di-Et phosphonate and probably VII. 80 g. Et-3-diethylphosphonopropionate, b.p. 114-15°, n_D²⁵ 1.4301, and 24.1 g. poly(ethyl acrylate), n_D²⁵ 1.3975. The acrylic monomer and polymer were identified by their infrared spectra.

Temperature rose slightly, and further heated for approx. 20 hrs. gave 33.6 g.

Et_{Br}; the mixture distilled in vacuo gave 43.9 g. of 1st fraction containing

diethyl phosphonate, VII, and unreacted material; the 2nd fraction was

contained 10.4 g. impure styrene. VII heated 5.5 hrs. at 211° and the material pumped down and heated gave no low-boiling materials. II (8.1 g.) was heated 2 hrs. at 210°/0.15 mm., without decomposition and heated again 4.5 hrs. at 250-305°/5 mm., still without decomposition II was recovered. Then the sample was heated at atmospheric pressure up to 390° to give a trace of H_2O and a viscous brown residue, which was strongly cicidic. The odor of styrene was strong in the trap but no styrene was isolated. The infrared spectrum showed polystyrene to be absent. III (10 g.) was dried overnight in vacuo, and heated 4 hrs. at 210-20°/0.15 mm.; the Dry Ice traps contained a small amount of liquid which had the odor of PhI and hinted at the presence of styrene.

Vapor phase chromatography showed the presence of a little styrene. The residue was crystallized to give II.

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LANGUAGE:	DOCUMENT NUMBER:	49:56835	CAPLUS		
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AB	TITLE:	Natoli, Giulio; Ercoli, Raffaele	"Montecatini" Societa Generale per l'Industria Mineraria e Chimica		
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The oxidation occurs on the C in the α -position to the substituted atom; there is formed an alc. of the structure $RCH(OH)CH_2CH_2O$. In order of decreasing ease of oxidation the radicals are CH_2 , Me, CH. The rates of oxidation are so different that only 1 of the possible alcs. forms if the radicals in the α -position are different. 2-Methyl-2-butene forms $Ba(OH)_2$ yields the alc., b160 136-8°. Likewise, 3-methyl-2-pentene forms 3-methyl-3-penten-2-ol, b18 54-6°. 2,3-Dimethyl-3-pentene (from the oxidation of 3-methyl-3-pentene by SeO₂ to 3-methyl-3-penten-2-ol, bromination by PbR₃ and reaction of the bromide with MeKBr), b760 91°, d421 0.720, mD21 1.4135, oxidizes to 2-isopropyl-2-butene-1-ol, b160 65-7°. 2,3,3-Trimethyl-3-pentene forms 2-tert-butyl-2-butene-1-ol, b1, b22 82°. 2-Methyl-2-pentene yields 2-methyl-2-penten-1-ol, b14 at 61-3°. 3-Phenyl-3-pentene forms 3-phenyl-3-penten-2-ol, b20 127-30°. 2-Cyclic hydrocarbons. It was observed that oxidation will occur in the α -position to the most substituted C atom and also in the cycle if it is possible. Oxidation of the ϵ -tpibond-CH group leads to conjugated dienes by way of the dehydration of tertiary alcs. Conjugated dienes also result from the oxidation of hydrocarbons possessing cyclic bi-tertiary double bonds. Thus, 1-ethylcyclohexene yields 30% of a liquid acetate of 1-ethylcyclohexen-6-ol which hydrolyzes to the corresponding alc., b12 82-83°. 1-Ethylcyclopentene forms 1-ethylcyclopenten-5-ol, b20 74-5°. 1,6-Dimethylcyclohexane oxidizes to 5 fractions, b. 127-8°, 130-5°, 132.5°, 134° and 135.5°. Each fraction contains o-xylene, a quantity of a liquid b160 132-5°, n_D²⁰ 1.4682, d₄₀ 0.832, and also 2,3-dimethyl-1,3-cyclohexadiene, identified by its maleic anhydride derivative, m. 122-3°. 1,2-Dimethylcyclohexene oxidizes to form 2 fractions. The 1st consists of o-xylene, and 2,3-dimethyl-1,3-cyclohexadiene, identified as above and also by its derivative with (ϵ -tpibond-COCMe)₂, b760 165-70°, which is hydrogenated to 4,5-dimethyl-1,2-phthalic acid, m. 196°. The 2nd fraction is a mixture of 2 acetates, one ethylenic and one dienic, yielding, on saponification, alc. products b23 95°, nD19 1.500, d419 0.971, but not in sufficient amts. to identify. III. Oxidation of hydrocarbons neither of whose ethylenic C atoms is completely substituted. 1. Aliphatic hydrocarbons. -Again, oxidation occurs in the α -position to the ethylenic C. The CH₂ radical oxidizes more readily than Me. A double bond at the end of the chain is as active as a bi-secondary bond due to transposition a primary alc. forms instead of a secondary alc. If a CH₂ radical is present on each side of the ethylenic C, both are oxidized and a mixture of alcs. forms when it is impossible to sep. Thus, 2-Pentene forms the acetate of 2-penten-4-ol which is saponified to the correspondin alc., b760 118-21°. 1-Hexene yields 2-hexen-1-ol, b160 156°, with a small amount of 1-hexen-3(?)-ol. 4-Nonenene on oxidation yields a liquid, b15 89-91°, which on saponification forms a viscous liqui b11 85-7°, and which is hydrogenated to a substance b18 90-1°. All attempts to prepare crystalline derivs. have failed. Presumably, the product is a mixture of nonenols formed by oxidation of the CH₂ groups in the α -position to the ethylenic C atoms. Likewise, 3-nonenene forms an acetate, b17 99-101°, which is saponified to nonenol, b15 93-5°, and hydrogenated to a mixture of nonanols, b17 93°. As in the preceding case no crystalline derivs. could be prepared. Cyclic hydrocarbons. While not so reactive as compds. with double-linked tertiary C atoms, cyclic hydrocarbons with bi-secondary ethylenic bonds do produce yields as high as 30-40%. Again the α -position is attacked. The CH₂ group is more active than the CH. Again, also, both possible CH₂ groups are attacked simultaneously. Transpositions of the allylpropenyllic type occur readily. Thus, cyclohexene yields the acetate of 1-octahexen-3-ol, b15 68-70°, saponifying to the corresponding alc., b15 67°, identified by its phenylurethane, m. 106 7-5°. Oxidation by CrO₃ forms the corresponding ketone whose semicarbazone m. 161°. 3-Methylcyclohexene likewise produces 6-methylcyclohexen-3-ol, b10 88-90°, which is hydrogenated to 4-methylcyclohexanol, b760

169°. Small amts. of MePh, 4-methylcyclohexene and 4-methylcyclohexen-3-ol also result from the oxidation of 4-methylcyclohexene. A mixture of the acetates of 4-, 5- and 6-methylcyclohexen-3-ol, b22 81-3°, the 1st predominating. The 3 corresponding alcs., formed on saponification, b63. Another fraction, b22 82-3°, is obtained which is also a mixture of the 3 acetates above but with the 5-Me derivative predominating. For all the oxidation products the author gives b. p. d. n. mol. refraction, as well as m. ps. of the various derivs. mentioned and in some cases C and H analyses. IV. Mechanism of the oxidation. The postulated mechanism may best be represented by means of the following equations in which R is a radical containing the ethylenic bond: (1) $4\text{RCH}_2\cdot\text{H} + \text{SeO}_2$
 $\rightarrow (\text{RCH}_2)_4\text{SE} + 2\text{H}_2\text{O}$ (2) $(\text{RCH}_2)_4\text{SE} + \text{H}_2\text{O} \rightarrow (\text{RCH}_2)_2\text{SE} + \text{RCH}_3 + (\text{RCH}_2)_3\text{OH}$ (3) $(\text{RCH}_2)_2\text{SE} + \text{H}_2\text{O} \rightarrow \text{CH}_2=\text{CHSE} + \text{RCH}_3 + \text{Se}$ This proposed mechanism explains (1) the fact that a portion of the original hydrocarbon is recovered although the theor. amount of SeO_2 is used, (2) the formation of an alkyl acetate even at low temps. since HOCH_2 may supplant OH in reaction 2 above, (3) the formation of an ether oxide in an absolute alc. medium by a mechanism similar to that which furnishes the acetate and (4) the formation of dienes with conjugated systems. This mechanism is supported by exptl. data on the oxidation of trimethylethylene in benzene. V. A complete table of the Raman spectra of all the compds. mentioned in the previous chapters is given, 38 compds. being listed in all.